



## Supporting Information

for

### **Continuous-flow carbonyl hydrogenation under subatmospheric to atmospheric hydrogen pressure enabled by robust heterogeneous Pt–Fe catalysts**

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**Additional experimental details, materials, and methods including photographs of reactor systems, STEM-EDS images and XPS spectra of heterogeneous catalysts, and copies of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra for isolated compounds**

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## General remarks

- NMR spectra were recorded with Bruker Avance NEO 400 MHz spectrometer operating at 400 MHz ( $^1\text{H}$ ) and 101 MHz ( $^{13}\text{C}$ ) respectively. Chemical shifts were recorded in parts per million (ppm), from relative to internal references of the  $\text{CDCl}_3$ , defined at 7.24 ppm ( $^1\text{H}$  NMR) and 77.0 ppm ( $^{13}\text{C}$  NMR) otherwise noted.
- GC analysis was performed on a Shimadzu GC-2030 apparatus with FID detector (column = Shimadzu SH-Polarwax, 0.25  $\mu\text{m}$ , 60 m;  $\text{N}_2$  gas pressure: 274.1 kPa, total flow: 144.3 mL/min, column flow: 3.01 mL/min, linear velocity: 42.5 cm/s, split ratio: 46.0, injection temperature: 250  $^\circ\text{C}$ , detector temperature: 280  $^\circ\text{C}$ ,  $\text{H}_2$  flow = 32.0 mL/min, air flow = 200 mL/min). Decane was used as internal standard for quantitative analysis.
- Inductively coupled plasma–atomic emission spectrometry (ICP–AES) analysis was performed on Horiba ULTIMA2 equipment.
- Aqua regia for ICP sample preparation was made by mixing concentrated hydrochloric acid and concentrated nitric acid in a volume of 3:1.
- Energy-dispersive X-ray spectroscopy/scanning transmission electron microscopes (STEM/EDS) images were obtained using a FEI JTECNAI OSIRIS instrument operated at 200 kV. All STEM specimens were prepared by placing a drop of the solution on carbon-coated copper grids and allowed to dry in air (without staining).
- X-ray photoelectron spectroscopy (XPS) was conducted by KRATOS ULTRA2 (Shimadzu). Binding energy was calibrated using internal carbon peak ( $\text{C}1\text{s} = 285.0$  eV).
- $\text{NaBH}_4$  was purchased from Fujifilm Wako Pure Chemical and it was stored and handled in a glove box.
- Organic reagents were used as purchased, or purified by distillation or recrystallization following the normal procedures if it is needed.

- Purchased from Fujifilm Wako Pure Chemical: **1b, 2b, 2e, 1g, 1h, 2h, 1i, 2i, 1j, 2j, 2m, 1g, 2g, 1r, 2u, 1w, 1x**. Purchased from TCI: **1a, 1c, 2c, 1d, 2d, 1e, 1f, 2f, 2g, 1k, 2k, 2k', 1l, 1m, 1n, 2n, 1p, 2p, 1o, 2o, 2r, 1s, 2s, 1t, 2t, 1u, 1v, 2v, 2w, 2x**. Purchased from Alfa: **2b**, purchased from Aldrich: **1v**. Purchased from BLD Pharmatech: **2v**
- Solvents and Celite® were purchased from Fujifilm Wako Pure Chemical. Deactivated glass wool (24324) was purchased from RESTEK.
- Pt/Al<sub>2</sub>O<sub>3</sub> (5 wt %) (Fujifilm Wako Pure Chemical), Pt/SiO<sub>2</sub> (5 wt %)(Strem), Pt/C (3 wt %)(Fujifilm Wako Pure Chemical), were used as commercially available catalysts.
- Na<sub>2</sub>PtCl<sub>6</sub>·6H<sub>2</sub>O was purchased from Aldrich. Pt black, PtO<sub>2</sub>, PtCl<sub>2</sub>, FeCl<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, Fe<sub>3</sub>O<sub>4</sub>, Fe(0), NiCl<sub>2</sub>·6H<sub>2</sub>O, and CoCl<sub>2</sub>·6H<sub>2</sub>O were purchased from Fujifilm Wako Pure Chemical
- Polysilane (DMPSi) was purchased from NIPPON SODA CO. LTD, and basic Al<sub>2</sub>O<sub>3</sub> was purchased from Merck. Pt/DMPSi-Al<sub>2</sub>O<sub>3</sub> and Pt-Au/DMPSi-Al<sub>2</sub>O<sub>3</sub> were prepared, following a method described in the literature <sup>1</sup>.

## Preparation of catalysts

### M/DMPSi–Al<sub>2</sub>O<sub>3</sub>

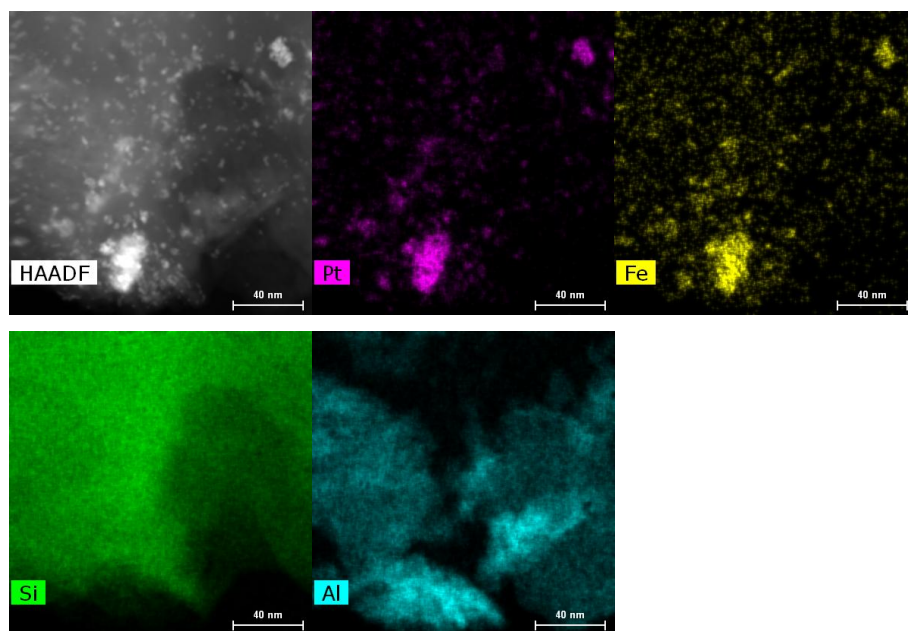
M<sup>1</sup>–M<sup>2</sup>/DMPSi–Al<sub>2</sub>O<sub>3</sub> catalysts were prepared, following procedures that have been reported in our previous article.<sup>1</sup> Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> catalysts with different Pt/Fe ratios were also prepared in a manner analogous to previously reported article.<sup>1</sup>

A representative procedure is given below.

#### **A procedure for the preparation of Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub>:**

To a suspension of polydimethylsilane (0.25 g) in THF (4 mL) was added NaBH<sub>4</sub> (81.4 mg) dissolved in diglyme (3 mL) at room temperature. Na<sub>2</sub>PtCl<sub>6</sub>·6H<sub>2</sub>O (67.4 mg) and FeCl<sub>2</sub> (15.8 mg) dissolved in THF (2 mL) was added dropwise to the solution at room temperature, and the reaction mixture was stirred at room temperature for 3 h. Then, basic alumina (1.25 g) was added to the mixture, which was then stirred at room temperature for 24 h. MeOH (200 mL) was added slowly to induce co-acervation, and the mixture was stirred at room temperature overnight. The resulting solid was collected by filtration and subsequently washed with a large excess of water until no brown solution came out from the filtrate. Afterward, it was washed with 100 mL of acetone, and the catalyst was kept wet with acetone to prevent the catalyst from burning. The collected solid was then dried in a vacuum at 100 °C for 3 h. Subsequently, the solid was heated to 150 °C under vacuum for 5 h. The solid was cooled under an argon atmosphere, after which the solid was washed with acetone (50 mL), water (50 mL), and CH<sub>2</sub>Cl<sub>2</sub> (50 mL) then dried in vacuo at 150 °C to afford the Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (1.5 g, Pt: 0.012 mmol/g Fe: 0.020 mmol/g).

### EDS mapping of Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Fe/Pt = 1.4):



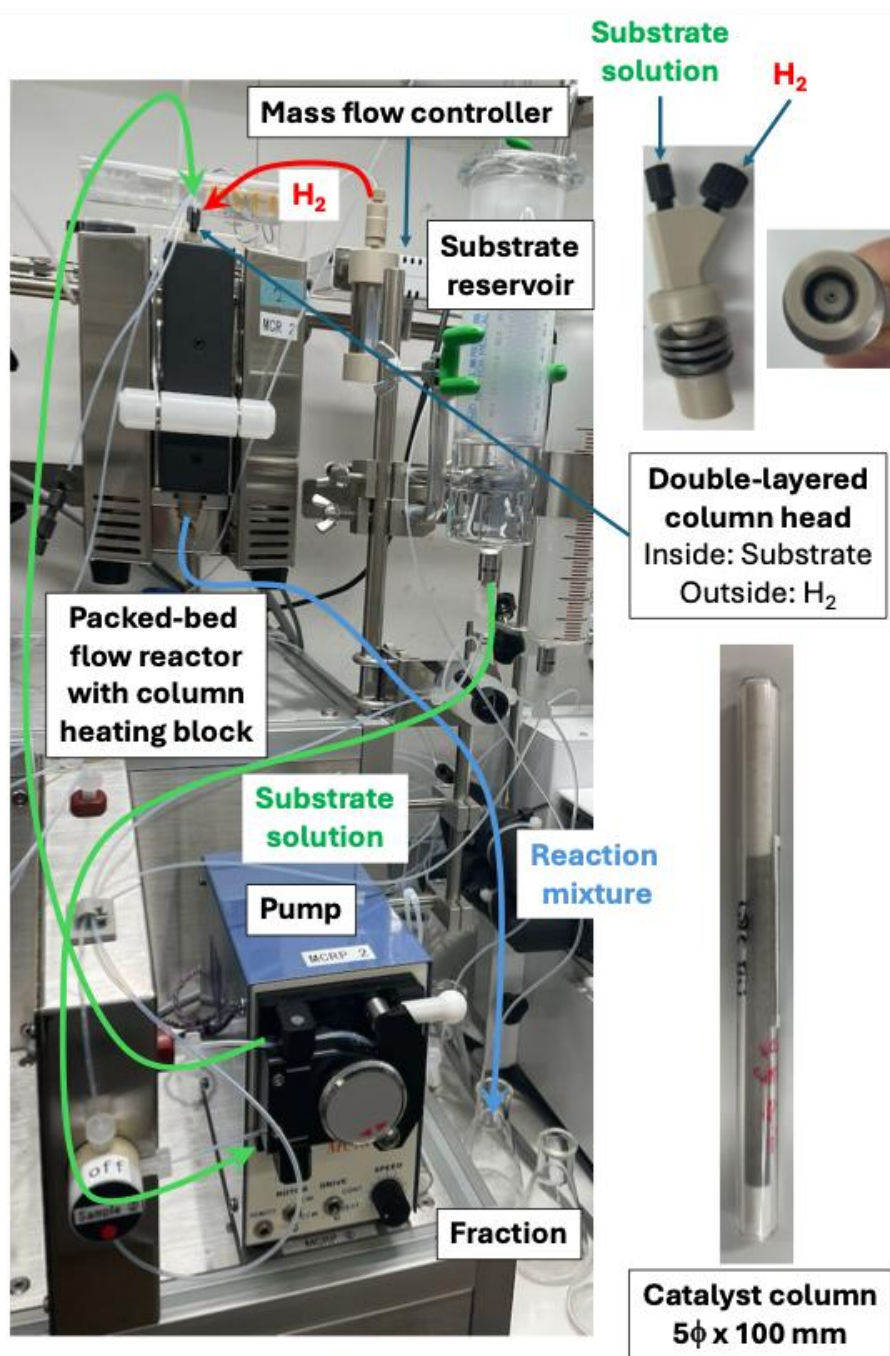
**Figure S1.** STEM-EDS mapping of Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub>

### Procedure to determine catalyst loading using ICP

Approximately 2–5 mg of catalyst was measured (with the amount measured being recorded) and placed in a test tube to which 1 mL of sulfuric acid was then added. The test tube was heated to 180 °C, and nitric acid was added dropwise until all solids had dissolved and no more brown fumes were observed. The mixture was cooled to room temperature and aqua regia (1 mL) was added slowly. The mixture was then treated by sonication until black precipitates were completely dissolved. The resulting mixture was brought up to a final volume of 50 mL with water in a volumetric flask, and the resulting solution was subjected to ICP analysis.

## Hydrogenation in continuous-flow system

### Reaction apparatus for hydrogenation



**Figure S2.** Photograph of flow reactor, double-layered column head and catalyst column

Continuous-flow reactions were conducted using an EYELA MCR-1000 column flow reactor (SynpleFlow™), a Minatoconcept MCRP204 peristaltic pump unit for introducing a liquid substrate, an EYELA MFC-11GU mass flow controller for introducing hydrogen gas (1–1.2 atm), and an EYELA MFC-1000 system that operates four individual mass flow controllers simultaneously for introducing mixed gas of H<sub>2</sub> and N<sub>2</sub>. Hydrogen gas was supplied by a PEAK Precision Hydrogen SL200 hydrogen gas generator. A 1/16-inch PTFE tube was used for all liquid and gas lines. Both gas and liquid were directed downward through a column head featuring a double-layered structure. The outer layer facilitated the introduction of gas, while the inner layer allowed for the introduction of liquid. Just before reaching the catalyst packing area, the two phases were thoroughly mixed through the use of a PTFE filter or glass wool filter.

### **Procedure for hydrogenation of carbonyl compounds under continuous-flow conditions**

In a manner analogous to our previous report,<sup>1</sup> heterogeneous catalyst (0.006 mmol as Pt) and Celite (0.5 g) were manually mixed in a glass vial through gentle shaking. The resulting mixed solid was then packed into a glass column, where the catalyst packing space measured 10 cm in length and had a 5 mm inner diameter for commercially available catalysts. The remaining space was filled with Celite. Pieces of glass wool were placed at both column ends, and the column was installed into the EYELA MCR-1000 column flow reactor (SynpleFlow™) with a double-layered column top unit for gas–liquid multi-phase reactions. To the column was fed 0.05 M of **1a** with decane (7–10 Mm, internal standard) in EtOAc using the Minatoconcept MCRP204 peristaltic pump unit (varied: 3.8–17 mL/h), and H<sub>2</sub> gas (fixed: 10 mL/min) was introduced simultaneously using the EYELA MFC-11GU mass flow controller in a downward flow direction. The flow rate was calculated by measuring the actually consumed volume of substrate solution in a fixed time. The flow reaction was conducted at room temperature to 80 °C, and the eluted solution was collected in a flask for a fixed period (every 30–120 min or 12 h (overnight running)). The continuous-flow reaction achieved a steady state after flowing under the same reaction conditions for more than 45

min. Following the continuous-flow of the substrate solution at a specific flow rate for a duration, the flow rate was subsequently adjusted to different values, and the resulting eluted solution was collected for analysis under the established steady state conditions.

Substrate solution includes internal standard (decane), and a collected fraction eluted from the flow reactor system was directly analyzed by GC-2030 (Shimadzu).

Isolation of products was conducted by simple evaporation of an eluted fraction and drying under vacuum. In this case, internal standard was not included in the solution. The structure of isolated compound was identified by comparison of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra with commercially available samples or already reported data in literature.

Details of the investigation using heterogeneous catalysts in Table 1 is summarized below.

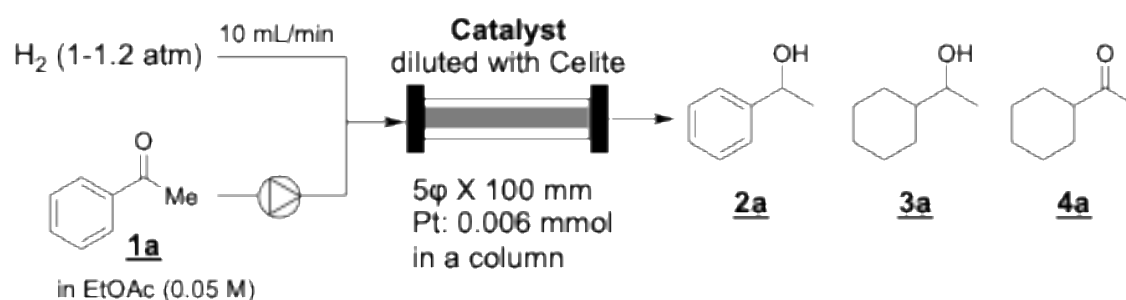
Flow rate of **1a** solution was gradually increased in time course, and results were collected at each flow rate.

**Definition of parameters:**

Yield = conversion × selectivity.

MHSV (molar hourly space velocity (/h)) = flow rate of substrate solution (L/h) × substrate concentration (M)/Pt (mol) in a column

TOF (turnover frequency of catalyst (/h)) = MHSV (/h) × yield



Representative results with different flow rates for each heterogeneous catalyst were summarized in tables below.

**Table S1.** Pt/C in a column

Flow rate (mL/h)	MHSV (/h)	<b>2a</b> (%) <sup>a</sup>	<b>3a</b> (%) <sup>a</sup>	<b>4a</b> (%) <sup>a</sup>	<b>1a</b> (%) <sup>a</sup>
4.0	33	16	2	10	72
7.6	63	7	1	6	83
9.4	78	4	0.4	3	92
13	125	3	0.3	2	95

<sup>a</sup> Determined by GC analysis with decane as internal standard

**Table S2.** Pt/SiO<sub>2</sub> in a column

Flow rate (mL/h)	MHSV (/h)	<b>2a</b> (%) <sup>a</sup>	<b>3a</b> (%) <sup>a</sup>	<b>4a</b> (%) <sup>a</sup>	<b>1a</b> (%) <sup>a</sup>
6.8	57	2	0	3	95
9.0	75	1	0	2	97
14	117	0.6	0	0.7	99

<sup>a</sup> Determined by GC analysis with decane as internal standard

**Table S3.** Pt/Al<sub>2</sub>O<sub>3</sub> in a column

Flow rate (mL/h)	MHSV (/h)	<b>2a</b> (%) <sup>a</sup>	<b>3a</b> (%) <sup>a</sup>	<b>4a</b> (%) <sup>a</sup>	<b>1a</b> (%) <sup>a</sup>
5.0	42	36	5	13	47
9.0	75	17	1	9	73
13.2	110	10	1	5	85
16	133	5	0	3	92

<sup>a</sup> Determined by GC analysis with decane as internal standard

**Table S4.** Pt/DMPSi–Al<sub>2</sub>O<sub>3</sub> in a column

Flow rate (mL/h)	MHSV (/h)	<b>2a</b> (%) <sup>a</sup>	<b>3a</b> (%) <sup>a</sup>	<b>4a</b> (%) <sup>a</sup>	<b>1a</b> (%) <sup>a</sup>
5.0	42	9	1	7	84
6.6	55	3	0	3	94
10.8	90	2	0	2	96
13	108	1	0	1	97

<sup>a</sup> Determined by GC analysis with decane as internal standard

**Table S5.** Pt–Au/DMPSi–Al<sub>2</sub>O<sub>3</sub> in a column

Flow rate (mL/h)	MHSV (/h)	<b>2a</b> (%) <sup>a</sup>	<b>3a</b> (%) <sup>a</sup>	<b>4a</b> (%) <sup>a</sup>	<b>1a</b> (%) <sup>a</sup>
4.2	35	38	3	12	47
7.2	60	20	1	8	71
11	92	10	1	5	84

<sup>a</sup> Determined by GC analysis with decane as internal standard

**Table S6.** Pt–Ni/DMPSi–Al<sub>2</sub>O<sub>3</sub> in a column

Flow rate (mL/h)	MHSV (/h)	<b>2a</b> (%) <sup>a</sup>	<b>3a</b> (%) <sup>a</sup>	<b>4a</b> (%) <sup>a</sup>	<b>1a</b> (%) <sup>a</sup>
5.4	45	32	2	0	66
9.2	77	14	1	0	85
14	117	7	0	0	93

<sup>a</sup> Determined by GC analysis with decane as internal standard

**Table S7.** Pt–Co/DMPSi–Al<sub>2</sub>O<sub>3</sub> in a column

Flow rate (mL/h)	MHSV (/h)	<b>2a</b> (%) <sup>a</sup>	<b>3a</b> (%) <sup>a</sup>	<b>4a</b> (%) <sup>a</sup>	<b>1a</b> (%) <sup>a</sup>
4.6	38	81	2	0	17
13	108	34	1	0	65
17	142	16	0	0	84

<sup>a</sup> Determined by GC analysis with decane as internal standard

**Table S8.** Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Fe/Pt = 0.62) in a column

Flow rate (mL/h)	MHSV (/h)	<b>2a</b> (%) <sup>a</sup>	<b>3a</b> (%) <sup>a</sup>	<b>4a</b> (%) <sup>a</sup>	<b>1a</b> (%) <sup>a</sup>
4.4	37	95	5	0	0
6.4	53	90	3	0	7
10.2	85	62	2	0	36
12.4	103	32	1	0	57
14.5	122	21	1	0	67

<sup>a</sup> Determined by GC analysis with decane as internal standard

**Table S9.** Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Fe/Pt = 1.4) in a column

Flow rate (mL/h)	MHSV (/h)	<b>2a</b> (%) <sup>a</sup>	<b>3a</b> (%) <sup>a</sup>	<b>4a</b> (%) <sup>a</sup>	<b>1a</b> (%) <sup>a</sup>
3.8	32	97	2	0	1
6.4	53	71	1	0	28
9.6	80	46	0	0	54
12	100	32	0	0	68

<sup>a</sup> Determined by GC analysis with decane as internal standard

**Table S10.** Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Fe/Pt = 2.3) in a column

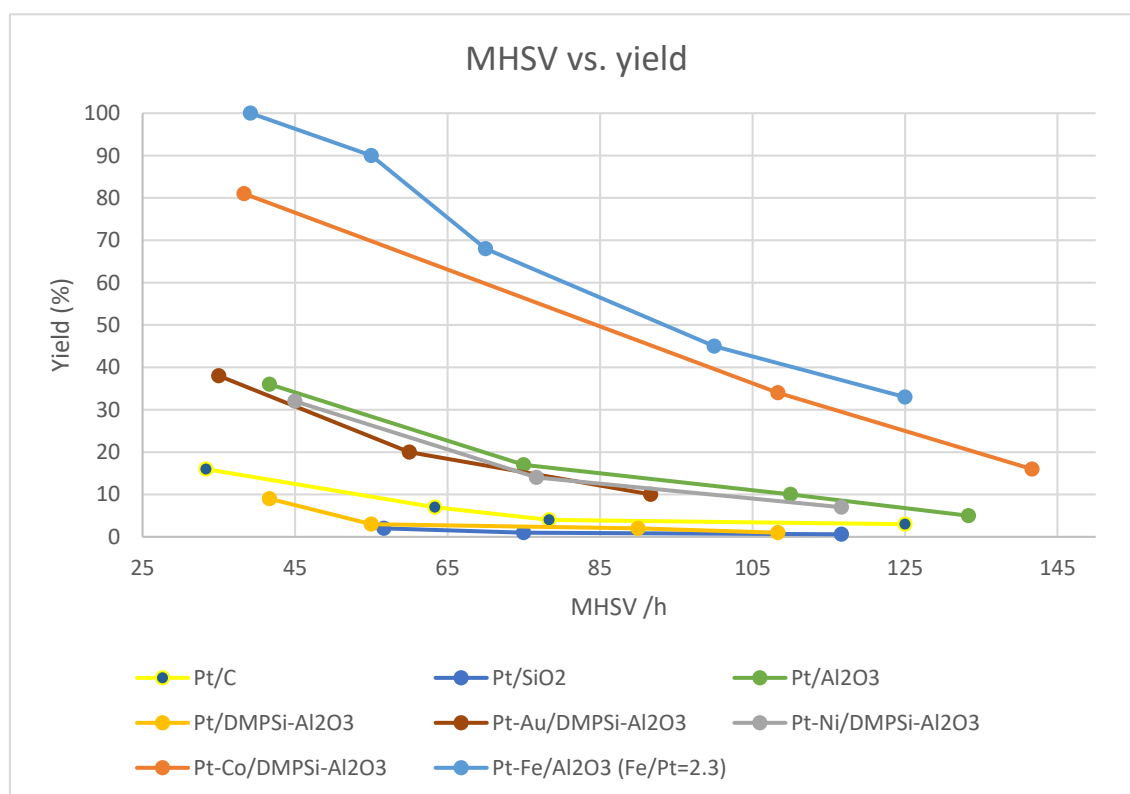
Flow rate (mL/h)	MHSV (/h)	<b>2a</b> (%) <sup>a</sup>	<b>3a</b> (%) <sup>a</sup>	<b>4a</b> (%) <sup>a</sup>	<b>1a</b> (%) <sup>a</sup>
4.7	39	>99	0	0	0
6.6	55	90	0	0	10
8.4	70	68	0	0	32
12	100	45	0	0	55
15	125	33	0	9	67

<sup>a</sup> Determined by GC analysis with decane as internal standard

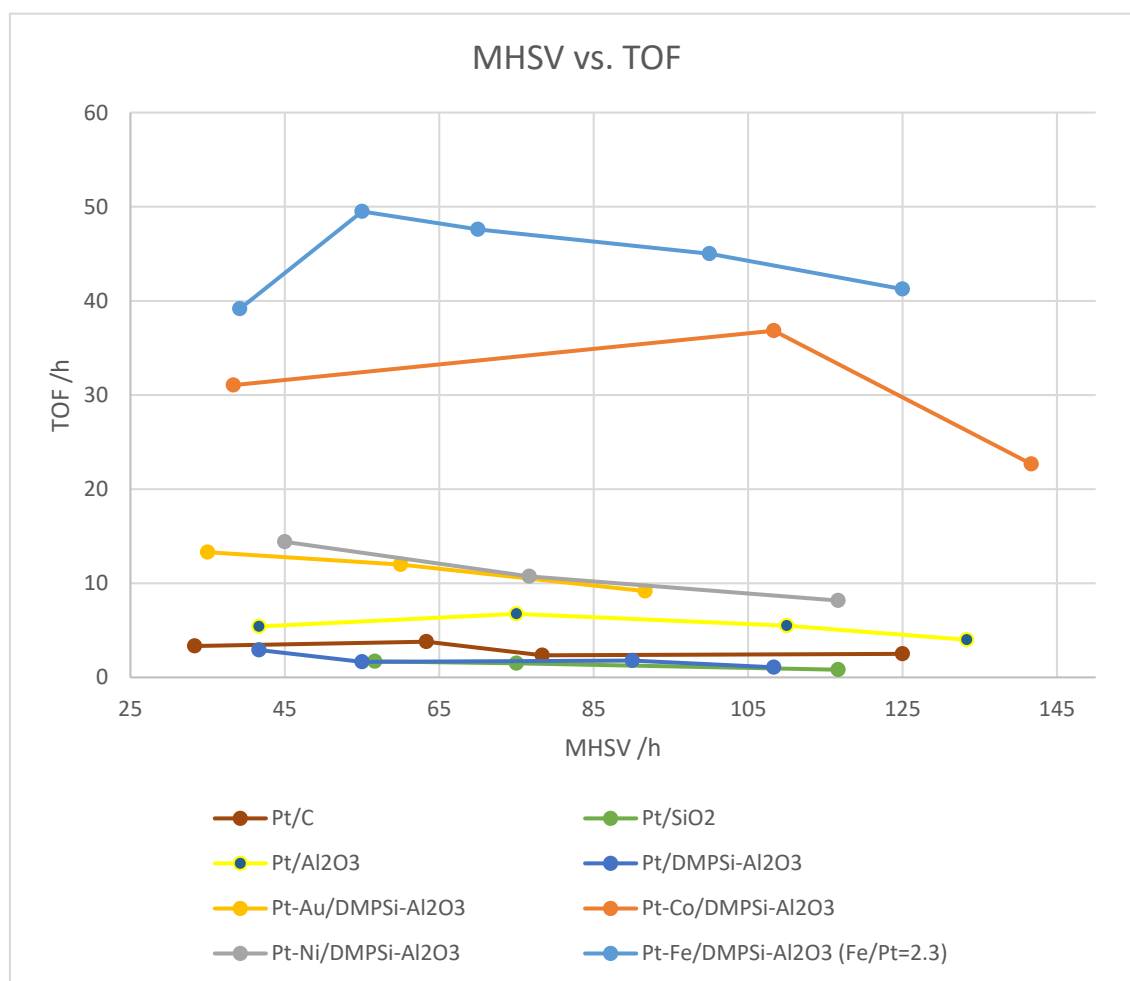
**Table S11.** Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Fe/Pt=4.4) in a column

Flow rate (mL/h)	MHSV (/h)	<b>2a</b> (%) <sup>a</sup>	<b>3a</b> (%) <sup>a</sup>	<b>4a</b> (%) <sup>a</sup>	<b>1a</b> (%) <sup>a</sup>
4.2	35	74	0	0	26
6.8	57	25	0	0	75
10.2	85	10	0	0	90
14.2	118	5	0	0	95

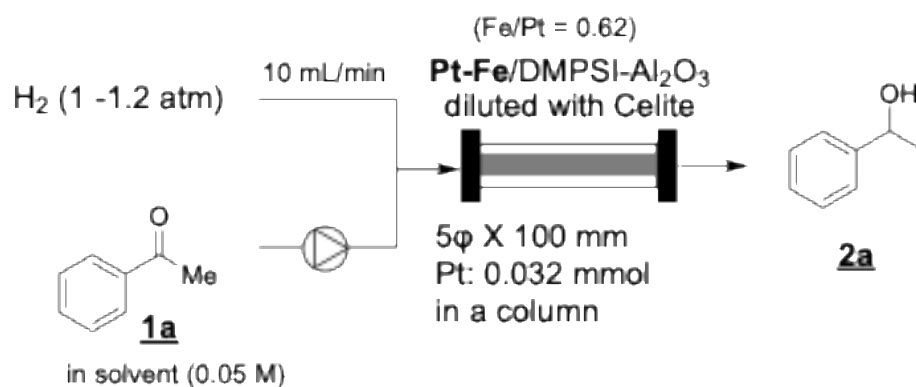
<sup>a</sup> Determined by GC analysis with decane as internal standard

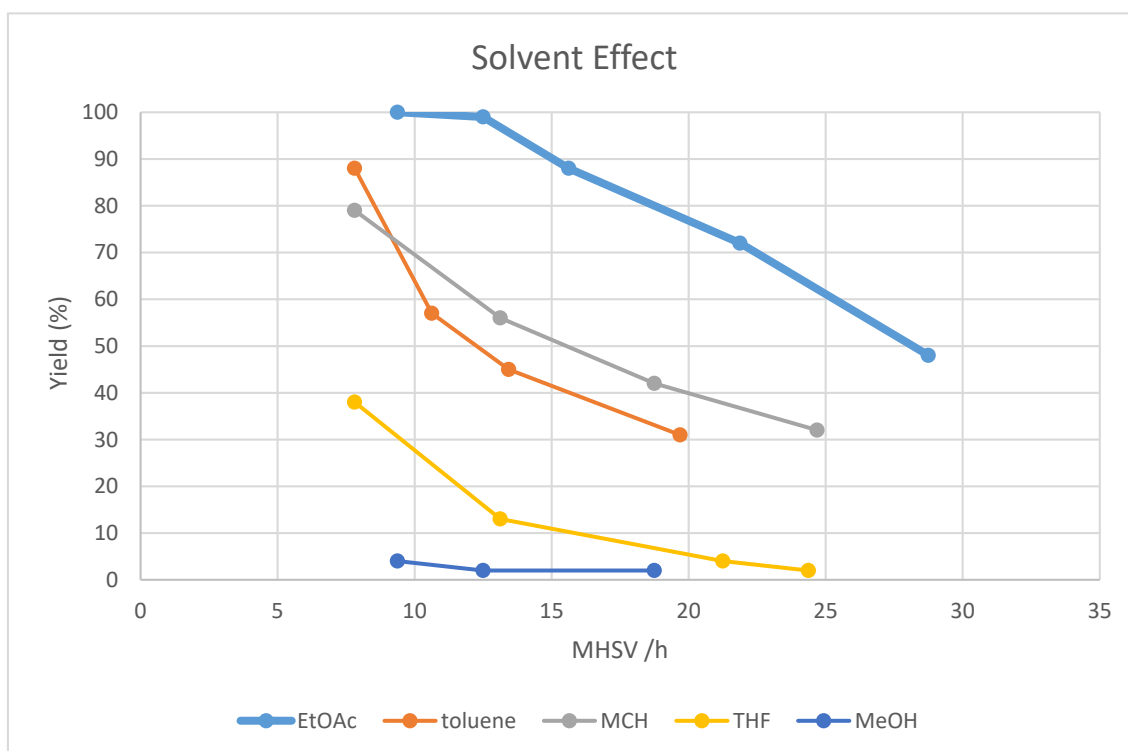


**Figure S3.** Comparison of catalysts (MHSV vs. yield)



**Figure S4.** Comparison of catalysts (MHSV vs. TOF).





**Figure S5.** Comparison of solvents (MHSV vs. Yield)

**Under sub-atmospheric hydrogen pressure conditions (Table 3, entry 1)**

Heterogeneous catalyst: Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.075 mmol/g Fe: 0.050 mmol/g, Fe/Pt = 0.67) 645 mg

Substrate solution: 0.05 M

H<sub>2</sub>: 0.2–0.24 atm, N<sub>2</sub>: 0.8–0.96 atm, total 20 mL/min

Flow rate (mL/h)	MHSV (/h)	<b>2a</b> (%) <sup>a</sup>	<b>1a</b> (%) <sup>a</sup>
3.8	3.9	>99	0
4.8	5.0	99	1
5.6	5.8	95	5
6.5	6.7	84	16
10.4	10.7	58	42

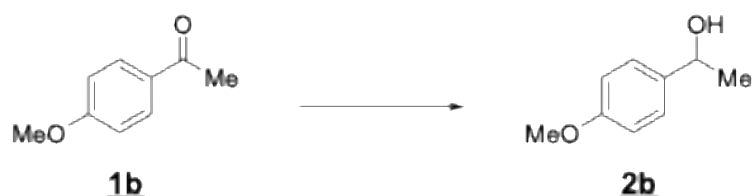
## Detailed results of substrate scope varying MHSV or isolation of products

### Hydrogenation of 1b

Heterogeneous catalyst: Pt-Fe/DMPsi-Al<sub>2</sub>O<sub>3</sub> (Pt: 0.075 mmol/g Fe: 0.050 mmol/g, Fe/Pt = 0.67) 435 mg

Substrate solution: 0.040 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min



Flow rate (mL/h)	MHSV (/h)	<b>2b</b> (%) <sup>a</sup>	<b>1b</b> (%) <sup>a</sup>
4.7	5.8	>99	0
7.2	8.8	91	9
11	13.5	70	30
13.3	16.3	52	48
16.6	20.4	38	61

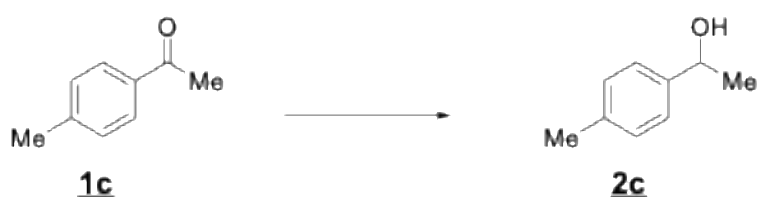
<sup>a</sup> Determined by GC analysis with decane as internal standard

### Hydrogenation of 1c

Heterogeneous catalyst: Pt-Fe/DMPsi-Al<sub>2</sub>O<sub>3</sub> (Pt: 0.075 mmol/g Fe: 0.050 mmol/g, Fe/Pt = 0.67) 430 mg

Substrate solution: 0.045 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min



Flow rate (mL/h)	MHSV (/h)	<b>2c</b> (%) <sup>a</sup>	<b>1c</b> (%) <sup>a</sup>
4.5	6.3	>99	0
7.0	9.8	94	6
11.7	16.3	79	21
13.4	18.7	69	31
15.4	21.5	56	44

<sup>a</sup> Determined by GC analysis with decane as internal standard

## Hydrogenation of **1d**

Heterogeneous catalyst: Pt-Fe/DMPSi-Al<sub>2</sub>O<sub>3</sub> (Pt: 0.075 mmol/g Fe: 0.050 mmol/g, Fe/Pt = 0.67) 430 mg

Substrate solution: 0.035 M

H<sub>2</sub>: 1-1.2 atm, 10 mL/min



At room temperature

Flow rate (mL/h)	MHSV (/h)	<b>2d</b> (%) <sup>a</sup>	<b>1d</b> (%) <sup>a</sup>
5.5	6.0	89	11
9.0	9.8	44	56
11.3	12.3	24	76
16.0	17.4	15	85
17.5	19.0	10	90

<sup>a</sup> Determined by GC analysis with decane as internal standard

At 50 °C

Flow rate (mL/h)	MHSV (/h)	<b>2d</b> (%) <sup>a</sup>	<b>1d</b> (%) <sup>a</sup>
5.4	5.9	>99	0
7.5	8.1	87	13
14.0	15.2	52	48
16.0	17.4	33	67
19.5	21.1	23	77

<sup>a</sup> Determined by GC analysis with decane as internal standard

Isolation of **2d**

Fixed flow rate: 3.8 mL/h at 50 °C

Fraction	Start time (h)	End time (h)	Yield <b>2d</b> (%) <sup>a</sup>
1	0	1.5	>99
2	1.5	3	>99
3	3	5	>99
4	5	8	>99
5	8	9	>99

<sup>a</sup> Isolated yield.

1-(2-Naphthyl)ethanol (**2d**) (identified by comparison with commercially available sample)

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.81 (ddd, *J* = 8.8, 5.1, 1.9 Hz, 4H), 7.54 – 7.41 (m, 3H), 5.05 (q, *J* = 6.4 Hz, 1H), 1.97 (s, 1H), 1.57 (d, *J* = 6.5 Hz, 3H).

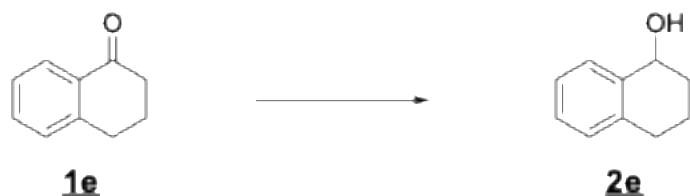
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 143.21, 133.36, 132.96, 128.33, 127.95, 127.69, 126.16, 125.81, 123.84, 123.82, 70.55, 25.14.

## Hydrogenation of **1e**

Heterogeneous catalyst: Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.075 mmol/g Fe: 0.050 mmol/g, Fe/Pt = 0.67) 430 mg

Substrate solution: 0.041 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min



Flow rate (mL/h)	MHSV (/h)	<b>2e</b> (%) <sup>a</sup>	<b>1e</b> (%) <sup>a</sup>
9.1	11.6	88	12
11.5	14.6	52	48
14.1	17.9	38	62
18.5	23.5	27	73

<sup>a</sup> Determined by GC analysis with decane as internal standard

## Hydrogenation of **1f**

Heterogeneous catalyst: Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.075 mmol/g Fe: 0.050 mmol/g, Fe/Pt = 0.67) 430 mg

Substrate solution: 0.031 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min

Fixed flow rate: 5.7 mL/h



Fraction	Start time (h)	End time (h)	Yield <b>2f</b> (%) <sup>a</sup>
1	0	1	>99
2	1	2	>99
3	2	3	>99
4	3	4	>99
5	4	5.5	>99
6	5.5	6.5	>99

<sup>a</sup> Isolated yield.

Ethyl 3-hydroxy-3-phenylpropanoate (**2f**) (identified by comparison with commercially available sample)

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.39 – 7.30 (m, 4H), 7.30 – 7.24 (m, 1H), 5.12 (dd, *J* = 8.7, 4.2 Hz, 1H), 4.17 (q, *J* = 7.1 Hz, 2H), 3.24 (s, 1H), 2.78 – 2.66 (m, 2H), 1.24 (t, *J* = 7.1 Hz, 3H).

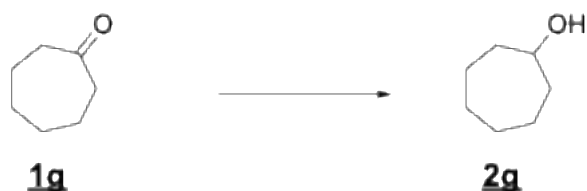
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 172.41, 142.53, 128.55, 127.80, 125.68, 70.35, 60.87, 43.35, 14.15.

## Hydrogenation of 1g

Heterogeneous catalyst: Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.014 mmol/g Fe: 0.020 mmol/g, Fe/Pt = 1.4) 428 mg

Substrate solution: 0.041 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min



Flow rate (mL/h)	MHSV (/h)	<b>2g</b> (%) <sup>a</sup>	<b>1g</b> (%) <sup>a</sup>
5.9	40.2	95	5
8.0	54.5	79	21
10.2	69.5	65	35
14.0	95.3	47	53

<sup>a</sup> Determined by GC analysis with decane as internal standard

## Hydrogenation of 1h

Heterogeneous catalyst: Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.075 mmol/g Fe: 0.050 mmol/g, Fe/Pt = 0.67) 430 mg

Substrate solution: 0.062 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min



Flow rate (mL/h)	MHSV (/h)	<b>2h</b> (%) <sup>a</sup>	<b>1h</b> (%) <sup>a</sup>
8.8	16.9	>99	0
11.0	21.1	>99	0
14.5	27.9	>99	0
18.2	35.0	>99	0

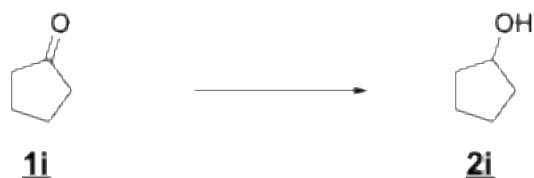
<sup>a</sup> Determined by GC analysis with decane as internal standard

## Hydrogenation of **1i**

Heterogeneous catalyst: Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.075 mmol/g Fe: 0.050 mmol/g, Fe/Pt = 0.67) 430 mg

Substrate solution: 0.072 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min



Flow rate (mL/h)	MHSV (/h)	<b>2i</b> (%) <sup>a</sup>	<b>1i</b> (%) <sup>a</sup>
7.3	16.3	98	2
11.4	25.5	84	16
13.8	30.8	67	33
19.1	42.6	47	53

<sup>a</sup> Determined by GC analysis with decane as internal standard

## Under sub-atmospheric hydrogen pressure conditions (Table 3, entries 2, 3)

Heterogeneous catalyst: Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.075 mmol/g Fe: 0.050 mmol/g, Fe/Pt = 0.67) 430 mg

Substrate solution: 0.072 M

H<sub>2</sub>: 0.2–0.24 atm, N<sub>2</sub>: 0.8–0.96 atm, total 20 mL/min

Flow rate (mL/h)	MHSV (/h)	<b>2i</b> (%) <sup>a</sup>	<b>1i</b> (%) <sup>a</sup>
6.9	15.4	97	3
8.0	17.9	96	4

Heterogeneous catalyst: Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.075 mmol/g Fe: 0.050 mmol/g, Fe/Pt = 0.67) 430 mg

Substrate solution: 0.072 M

H<sub>2</sub>: 0.1–0.12 atm, N<sub>2</sub>: 0.9–1.08 atm, total 20 mL/min

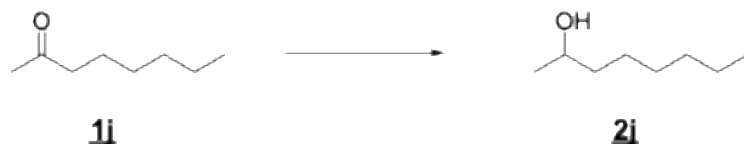
Flow rate (mL/h)	MHSV (/h)	<b>2i</b> (%) <sup>a</sup>	<b>1i</b> (%) <sup>a</sup>
6.3	14.1	84	16

## Hydrogenation of 1j

Heterogeneous catalyst: Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.075 mmol/g Fe: 0.050 mmol/g, Fe/Pt = 0.67) 430 mg

Substrate solution: 0.047 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min



Flow rate (mL/h)	MHSV (/h)	<b>2j</b> (%) <sup>a</sup>	<b>1j</b> (%) <sup>a</sup>
4.7	6.8	>99	0
8.8	12.8	91	9
13.7	20.0	89	11
15.8	23.0	71	29
19.5	28.4	51	49

<sup>a</sup> Determined by GC analysis with decane as internal standard

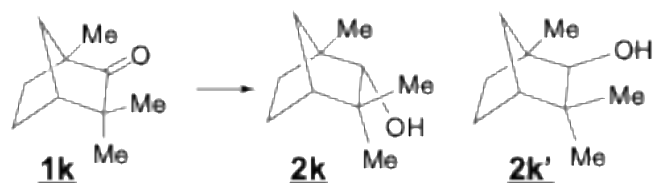
## Hydrogenation of 1k

Heterogeneous catalyst: Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.025 mmol/g Fe: 0.058 mmol/g, Fe/Pt = 2.3) 240 mg

Substrate solution: 0.016 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min

Fixed flow rate: 4.3 mL/h, MHSV: 12 /h



Temperature °C	Yield of <b>2k</b> (%)	Yield of <b>2k'</b> (%)
50	3	14
60	5	18
70	19	48
80	25	49
90	21	41

<sup>a</sup> Determined by GC analysis with decane as internal standard

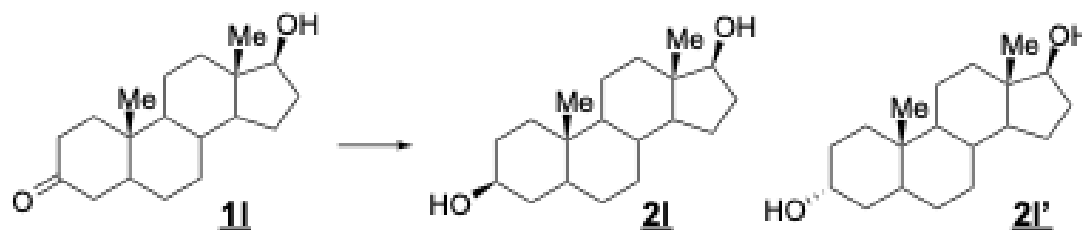
## Hydrogenation of **11**

Heterogeneous catalyst: Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.025 mmol/g Fe: 0.058 mmol/g, Fe/Pt = 2.3) 240 mg

Substrate solution: 0.01 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min

Fixed flow rate: 4.7 mL/h, MHSV: 8 /h



Fraction	Start time (h)	End time (h)	Yield <b>21</b> (%) <sup>a</sup>	Yield <b>21'</b> (%) <sup>a</sup>
1	0	1.5	77	23
2	1.5	2.5	75	25
3	2.5	4.5	74	26
4	4.5	6	74	26
5	6	8	74	26
6	8	9	74	26

<sup>a</sup> Obtained as mixture of isomers. Ratio of isomers was determined by <sup>1</sup>H NMR analysis<sup>2</sup>.

Major isomer (**21**) <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 3.56 – 3.65 (m, 2H), 2.00 – 2.10 (m, 1H), 1.64 – 1.85 (m, 4H), 1.20 – 1.50 (m, 12H), 0.85 – 1.20 (m, 6H), 0.82 (3H, s), 0.73 (3H, s), δ 0.63 (ddd, *J* = 12.3, 10.4, 4.1 Hz, 1H).<sup>2</sup>

## Hydrogenation of 1m

Heterogeneous catalyst: Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.014 mmol/g Fe: 0.020 mmol/g, Fe/Pt = 0.67) 428 mg

Substrate solution: 0.057 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min



**1m**

**2m**

Flow rate (mL/h)	MHSV (/h)	2m (%) <sup>a</sup>	1m (%) <sup>a</sup>
7.1	67.5	98	2
9.0	85.6	98	2
10.0	95.1	99	1
13.6	129.4	99	1
18.9	179.8	>99	0

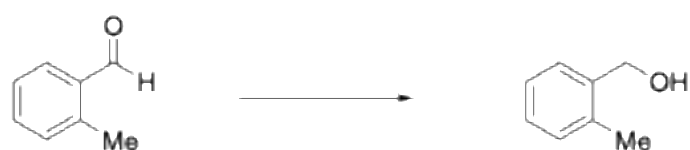
<sup>a</sup> Determined by GC analysis with decane as internal standard

## Hydrogenation of 1n

Heterogeneous catalyst: Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.014 mmol/g Fe: 0.020 mmol/g, Fe/Pt = 0.67) 428 mg

Substrate solution: 0.05 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min



**1n**

**2n**

Flow rate (mL/h)	MHSV (/h)	2n (%) <sup>a</sup>	1n (%) <sup>a</sup>
6.0	50.1	98	2
8.7	72.6	>99	0
12.8	106.8	91	9
15.0	125.2	83	17

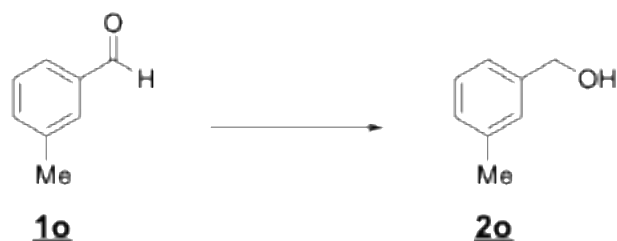
<sup>a</sup> Determined by GC analysis with decane as internal standard

## Hydrogenation of 1o

Heterogeneous catalyst: Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.014 mmol/g Fe: 0.020 mmol/g, Fe/Pt = 0.67) 428 mg

Substrate solution: 0.05 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min



Flow rate (mL/h)	MHSV (/h)	<b>2o</b> (%) <sup>a</sup>	<b>1o</b> (%) <sup>a</sup>
6.0	50.1	>99	0
8.4	70.1	>99	0
12.8	106.8	98	2
16.5	137.7	93	7
19.7	164.4	87	13

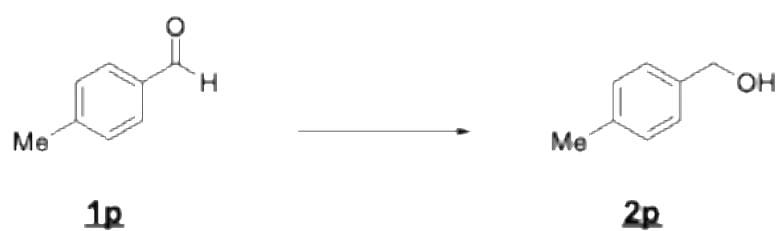
<sup>a</sup> Determined by GC analysis with decane as internal standard

## Hydrogenation of 1p

Heterogeneous catalyst: Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.014 mmol/g Fe: 0.020 mmol/g, Fe/Pt = 0.67) 428 mg

Substrate solution: 0.05 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min



Flow rate (mL/h)	MHSV (/h)	<b>2p</b> (%) <sup>a</sup>	<b>1p</b> (%) <sup>a</sup>
5.7	47.6	>99	0
9.0	75.1	>99	0
12.5	104.3	95	5
16.4	136.8	86	14
20.9	174.4	74	26

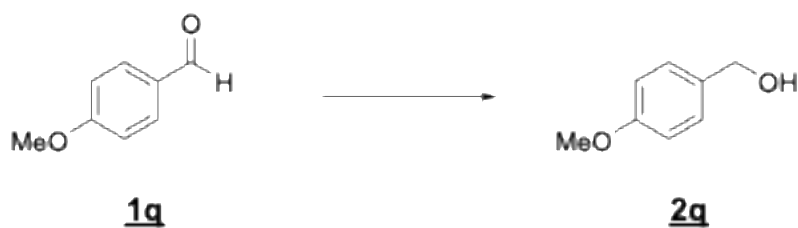
<sup>a</sup> Determined by GC analysis with decane as internal standard

## Hydrogenation of 1q

Heterogeneous catalyst: Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.014 mmol/g Fe: 0.020 mmol/g, Fe/Pt = 0.67) 428 mg

Substrate solution: 0.03 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min



Flow rate (mL/h)	MHSV (/h)	<b>2q</b> (%) <sup>a</sup>	<b>1q</b> (%) <sup>a</sup>
5.4	27.0	98	2
7.8	39.1	>99	0
9.9	49.6	>99	0
13.8	69.1	>99	0
17.7	88.6	>99	0

<sup>a</sup> Determined by GC analysis with decane as internal standard

## Hydrogenation of 1r

Heterogeneous catalyst: Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.014 mmol/g Fe: 0.020 mmol/g, Fe/Pt = 0.67) 428 mg

Substrate solution: 0.043 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min



Flow rate (mL/h)	MHSV (/h)	<b>2r</b> (%) <sup>a</sup>	<b>1r</b> (%) <sup>a</sup>
5.8	41.6	94	0
10.2	73.2	96	0
13.3	95.4	97	0
18.2	130.6	98	0

<sup>a</sup> Determined by GC analysis with decane as internal standard

## Hydrogenation of **1s**

Heterogeneous catalyst: Pt–Fe/DMPsi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.075 mmol/g Fe: 0.050 mmol/g, Fe/Pt = 0.67) 430 mg

Substrate solution: 0.038 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min



Flow rate (mL/h)	MHSV (/h)	<b>2s</b> (%) <sup>a</sup>	<b>1s</b> (%) <sup>a</sup>
5.1	6.0	>99	0
7.6	9.0	>99	0
12.9	15.2	55	45
14.0	16.5	36	64

<sup>a</sup> Determined by GC analysis with decane as internal standard

## Isolation of **2s**

Fixed flow rate: 6.8 mL/h at room temperature

Fraction	Start time (h)	End time (h)	Yield <b>2t</b> (%) <sup>a</sup>
1	0	0.83	>99
2	0.83	3.1	>99
3	3.1	4.1	98
4	4.1	5.4	>99
5	5.4	6.5	97
6	6.5	7.5	>99

<sup>a</sup> Isolated yield.

Naphthalen-2-ylmethanol (**2s**) (identified by comparison with commercially available sample)

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.81 (ddd, *J* = 10.6, 6.0, 2.5 Hz, 4H), 7.58 – 7.39 (m, 3H), 4.84 (d, *J* = 5.4 Hz, 2H), 1.81 (t, *J* = 5.8 Hz, 1H).

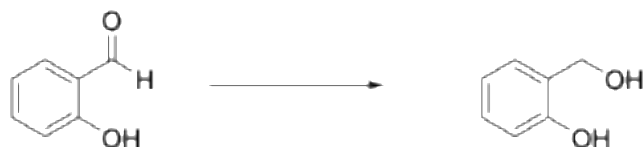
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 138.33, 133.41, 132.98, 128.36, 127.90, 127.73, 126.20, 125.92, 125.46, 125.17, 65.51.

## Hydrogenation of **1t**

Heterogeneous catalyst: Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.075 mmol/g Fe: 0.050 mmol/g, Fe/Pt = 0.67) 430 mg

Substrate solution: 0.05 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min



**1t**

**2t**

Flow rate (mL/h)	MHSV (/h)	<b>2t</b> (%) <sup>a</sup>	<b>1t</b> (%) <sup>a</sup>
5.2	8.1	>99	0
8.7	13.5	>99	0
12.2	18.9	86	14
16.6	25.7	67	33
18.0	27.9	55	45

<sup>a</sup> Determined by GC analysis with decane as internal standard

## Isolation of **2t**

Fixed flow rate: 6 mL/h at room temperature

Fraction	Start time (h)	End time (h)	Yield <b>2t</b> (%) <sup>a</sup>
1	0	0.67	>99
2	0.67	1.67	>99
3	1.67	2.5	>99
4	2.5	3.75	>99
5	3.75	5.5	>99
6	5.5	6.5	>99

<sup>a</sup> Isolated yield.

2-(Hydroxymethyl)phenol (**2t**) (identified by comparison with commercially available sample)

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.3 (br 1H), 7.19 (td, *J* = 7.7, 1.7 Hz, 1H), 7.02 (dd, *J* = 7.5, 1.7 Hz, 1H), 6.92 – 6.76 (m, 2H), 4.83 (s, 2H), 2.42 (br, 1H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 156.02, 129.52, 127.88, 124.72, 120.13, 116.53, 64.58.

## Hydrogenation of 1u

Heterogeneous catalyst: Pt–Fe/DMPsi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.075 mmol/g Fe: 0.050 mmol/g, Fe/Pt = 0.67) 430 mg

Substrate solution: 0.057 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min



Flow rate (mL/h)	MHSV (/h)	<b>2u</b> (%) <sup>a</sup>	<b>1u</b> (%) <sup>a</sup>
5.3	9.4	91	9
6.9	12.2	22	78
11.0	19.4	10	90
14.0	24.7	4	96

<sup>a</sup> Determined by GC analysis with decane as internal standard

## Hydrogenation of 1v

Heterogeneous catalyst: Pt–Fe/DMPsi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.075 mmol/g Fe: 0.050 mmol/g, Fe/Pt = 0.67) 430 mg

Substrate solution: 0.053 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min



Flow rate (mL/h)	MHSV (/h)	<b>2v</b> (%) <sup>a</sup>	<b>1v</b> (%) <sup>a</sup>
5.2	8.5	61	39
7.5	12.3	29	71
12.4	20.4	18	82
15.6	25.6	13	87
17.8	29.3	11	89

<sup>a</sup> Determined by GC analysis with decane as internal standard

## Hydrogenation of 1w

Heterogeneous catalyst: Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.075 mmol/g Fe: 0.050 mmol/g, Fe/Pt = 0.67) 430 mg

Substrate solution: 0.047 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min



Flow rate (mL/h)	MHSV (/h)	<b>2w</b> (%) <sup>a</sup>	<b>1w</b> (%) <sup>a</sup>
8.2	12.0	98	2
11.3	16.5	97	3
15.0	21.9	96	4
16.7	24.3	97	3

<sup>a</sup> Determined by GC analysis with decane as internal standard

## Under sub-atmospheric hydrogen pressure conditions (Table 3, entry 5)

Heterogeneous catalyst: Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.061 mmol/g Fe: 0.038 mmol/g, Fe/Pt = 0.62) 100 mg

Substrate solution: 0.032 M

H<sub>2</sub>: 0.2–0.24 atm, N<sub>2</sub>: 0.8–0.96 atm, total 10 mL/min

Flow rate (mL/h)	MHSV (/h)	<b>2w</b> (%) <sup>a</sup>	<b>1w</b> (%) <sup>a</sup>
4.0	21.0	95	0
5.9	31.0	90	9
8.8	46.1	75	25
12.0	63.0	46	54

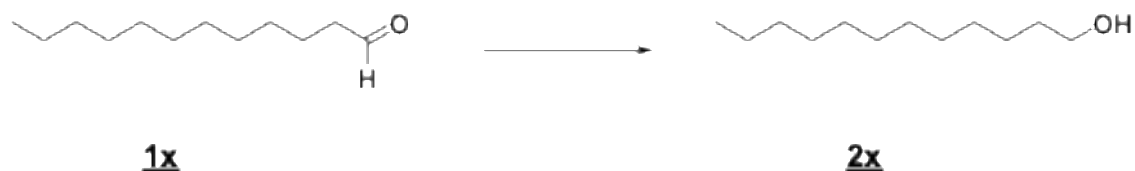
## Hydrogenation of **1x**

Heterogeneous catalyst: Pt–Fe/DMPsi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.075 mmol/g Fe: 0.050 mmol/g, Fe/Pt = 0.67) 430 mg

Substrate solution: 0.033 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min

Fixed flow rate: 10.4 mL/h at room temperature



Fraction	Start time (h)	End time (h)	Yield <b><u>2x</u></b> (%) <sup>a</sup>
1	0	0.75	>99
2	0.75	1.75	>99
3	1.75	2.75	>99
4	2.75	3.75	>99
5	3.75	5.25	>99
6	5.25	6.75	>99
7	6.75	7.75	>99
8	7.75	9	>99

<sup>a</sup> Isolated yield.

1-Dodecanol (**2x**) (identified by comparison with commercially available sample)

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 3.61 (t, *J* = 6.6 Hz, 2H), 1.53 (dt, *J* = 8.1, 6.4 Hz, 2H), 1.37 – 1.15 (m, 19H), 0.91 – 0.81 (m, 3H).

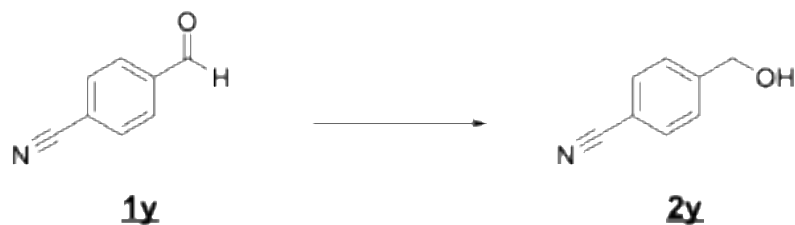
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 63.08, 32.82, 31.91, 29.66, 29.63, 29.61, 29.59, 29.44, 29.34, 25.75, 22.68, 14.09.

## Hydrogenation of **1y**

Heterogeneous catalyst: Pt–Fe/DMPSi–Al<sub>2</sub>O<sub>3</sub> (Pt: 0.036 mmol/g Fe: 0.044 mmol/g, Fe/Pt = 1.2) 167 mg

Substrate solution: 0.03 M

H<sub>2</sub>: 1–1.2 atm, 10 mL/min

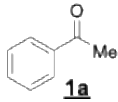
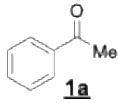
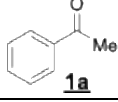
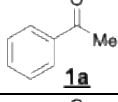
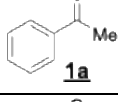
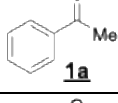
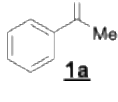
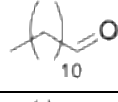
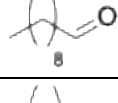
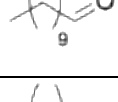
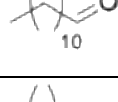
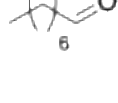


Flow rate (mL/h)	MHSV (/h)	<b>2y</b> (%) <sup>a</sup>	<b>1y</b> (%) <sup>a</sup>
7.0	34.9	95	0
14.0	70.0	81	19
18.0	89.8	56	44

<sup>a</sup> Determined by GC analysis with decane as internal standard

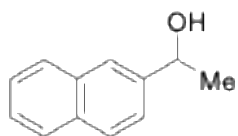
## Comparison of heterogeneous catalysts for hydrogenation of carbonyl compounds

**Table S12.** Comparison of heterogeneous catalysts for hydrogenation of carbonyl compounds

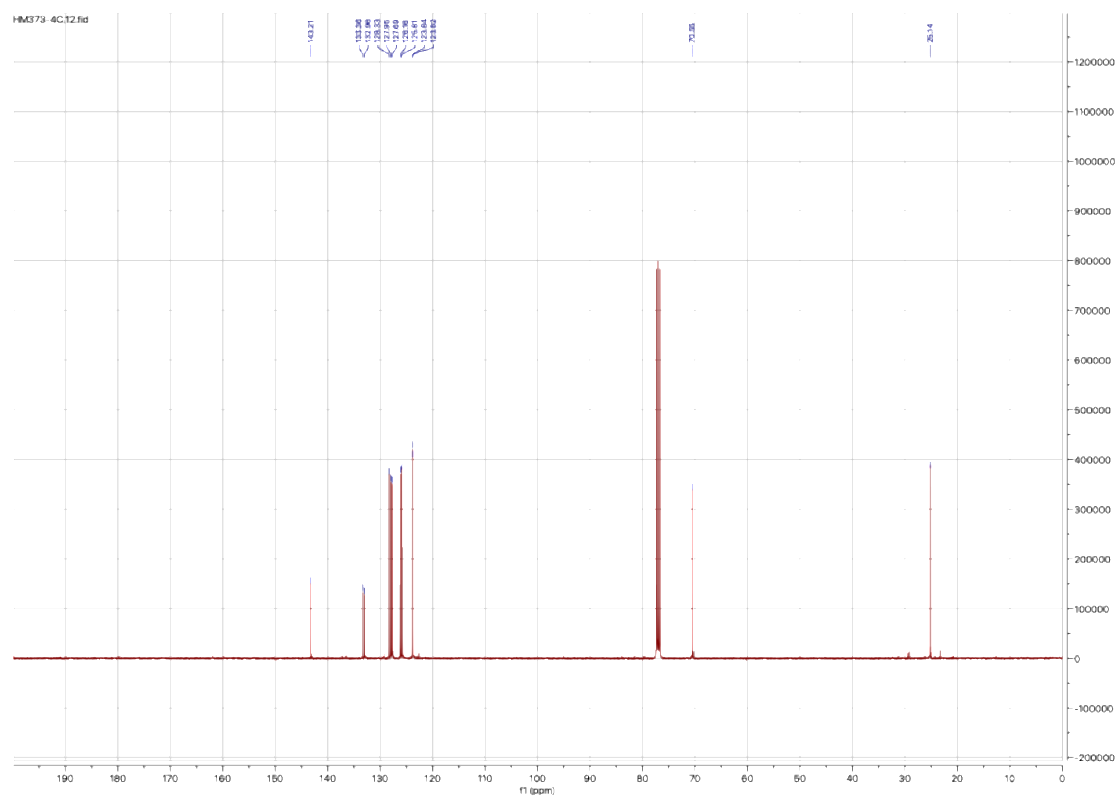
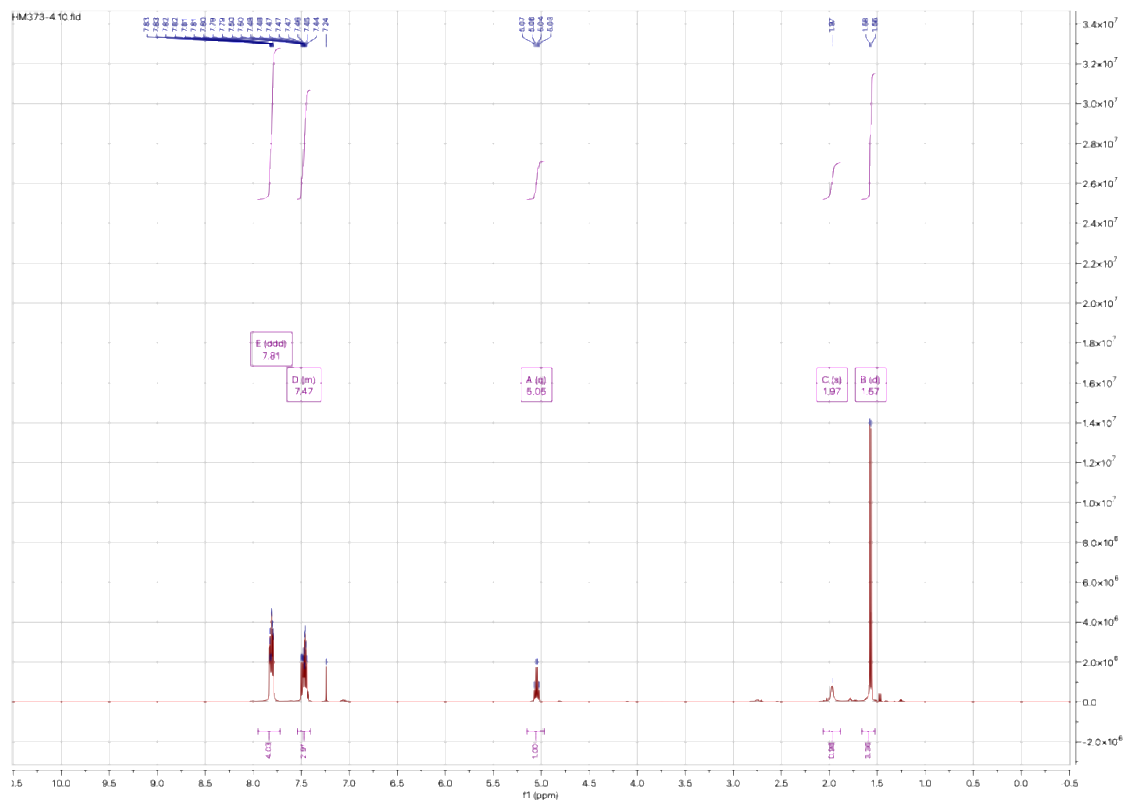
Catalyst	substrate	Temperature	H <sub>2</sub> pressure	Yield	Conversion	Selectivity	Reference
Pt/Reduced graphene oxide		40 °C	40 atm	86%	>99%	86%	Ref. 3
ARP-Pt		40 °C	1 atm	52%	>99%	52%	Ref. 4
Ru/FCN		40 °C	1 atm	61%	79%	77%	Ref. 5
Ru-Pt/PVP		30 °C	200 KPa	71%	99%	72%	Ref. 6
Plated milling vessel		rt	5 bar	51%	–	–	Ref. 7
<b>Pt-Fe/DMPSi-Al<sub>2</sub>O<sub>3</sub></b>		rt	<b>1 atm (101 KPa)</b>	<b>&gt;99%</b>	<b>&gt;99%</b>	<b>&gt;99%</b>	<b>This work</b>
<b>Pt-Fe/DMPSi-Al<sub>2</sub>O<sub>3</sub></b>		rt	<b>0.24 atm (24 KPa)</b>	<b>93%</b>	<b>93%</b>	<b>&gt;99%</b>	<b>This work</b>
Ru-Pt/PVP		30 °C	100 KPa	52%	-	-	Ref. 6
ARP-Pt		40 °C	1 atm	17%	43%	40%	Ref. 4
Plated milling vessel		rt	5 bar	43%	-	-	Ref. 7
<b>Pt-Fe/DMPSi-Al<sub>2</sub>O<sub>3</sub></b>		rt	<b>1 atm (101 kPa)</b>	<b>&gt;99%</b>	<b>&gt;99%</b>	<b>&gt;99%</b>	<b>This work</b>
<b>Pt-Fe/DMPSi-Al<sub>2</sub>O<sub>3</sub></b>		rt	<b>0.24 atm (24 KPa)</b>	<b>97%</b>	<b>97%</b>	<b>&gt;99%</b>	<b>This work</b>

# $^1\text{H}$ and $^{13}\text{C}$ NMR spectra of isolated compounds

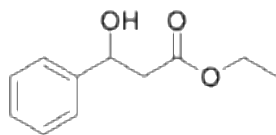
## 1-(2-Naphtyl)ethanol (2d)



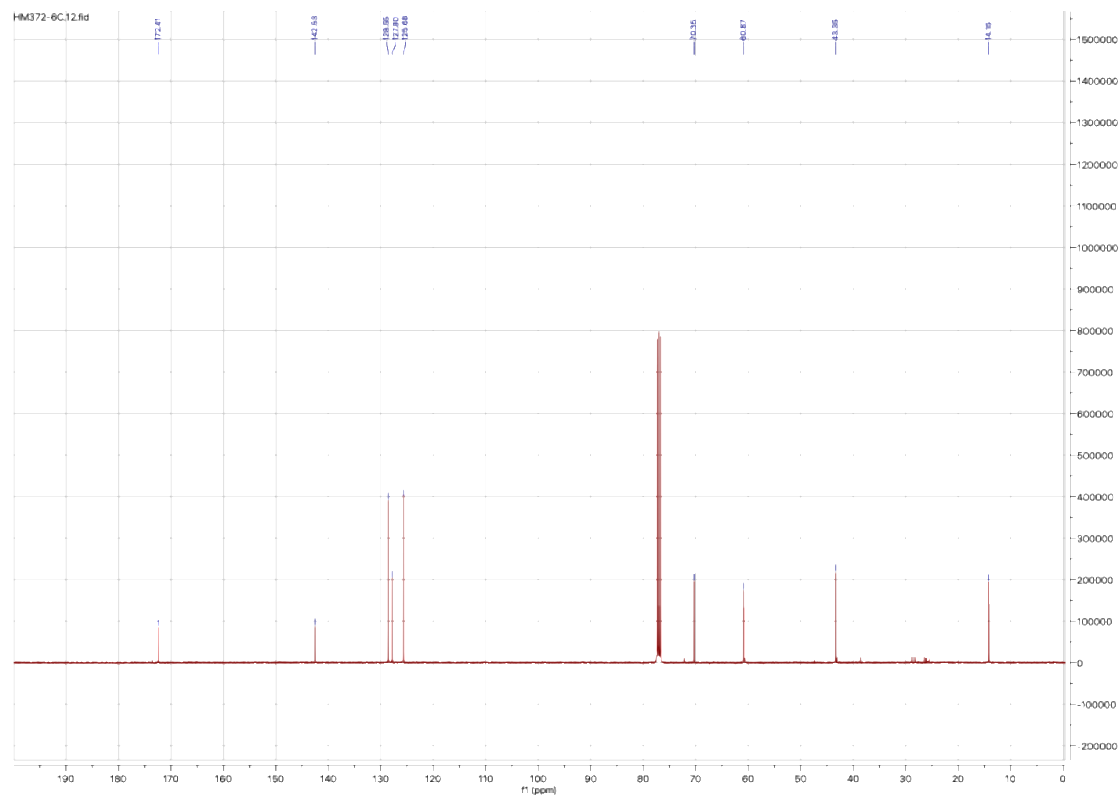
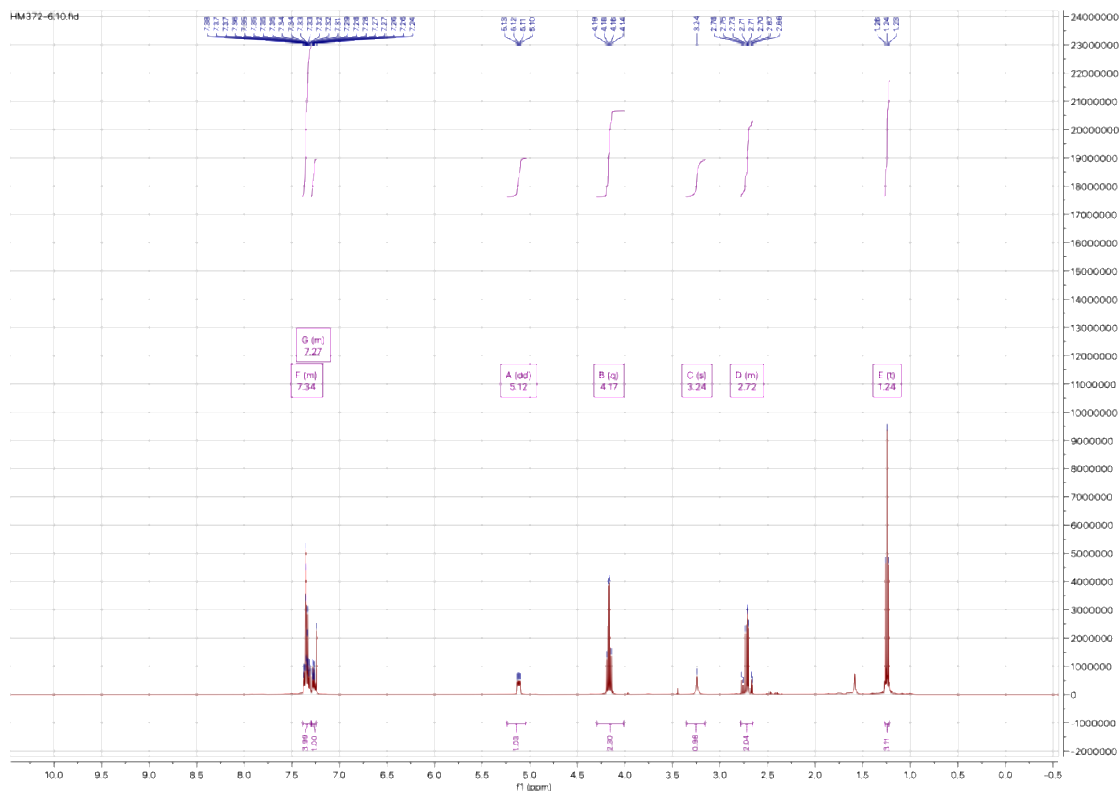
**2d**



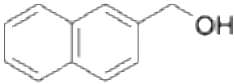
# Ethyl 3-hydroxy-3-phenylpropanoate (2f)



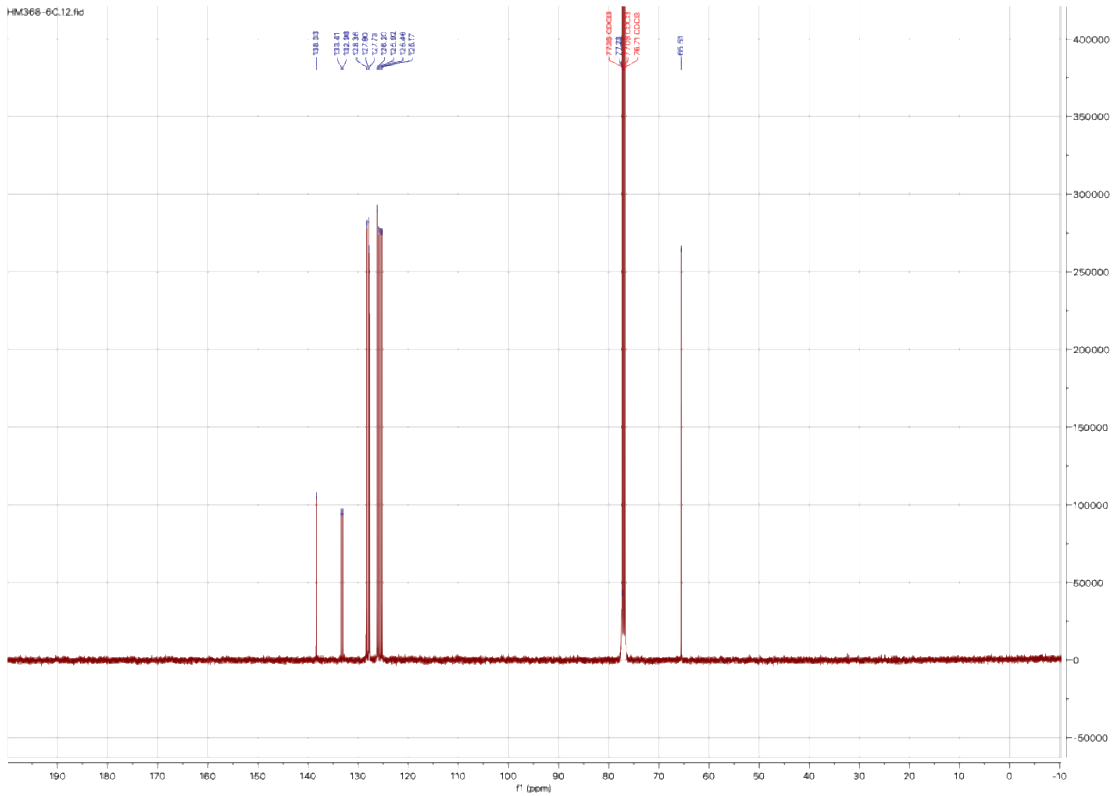
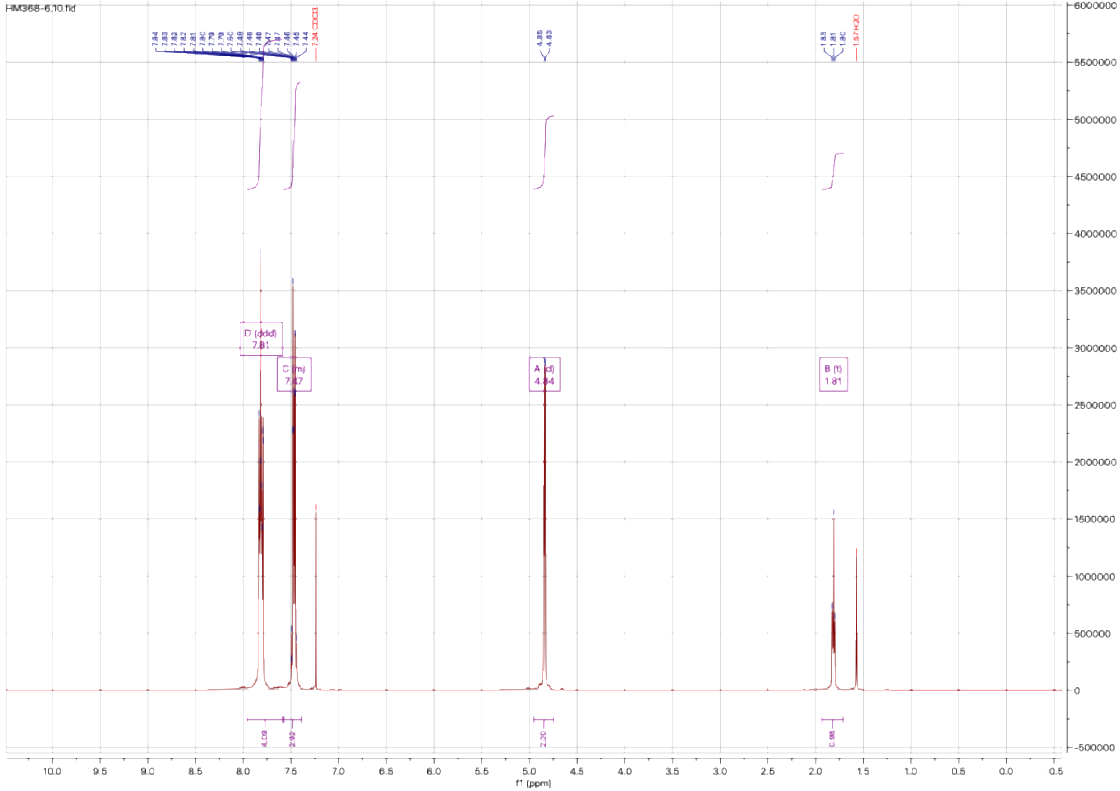
**2f**



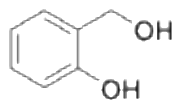
# Naphthalen-2-ylmethanol (2s)



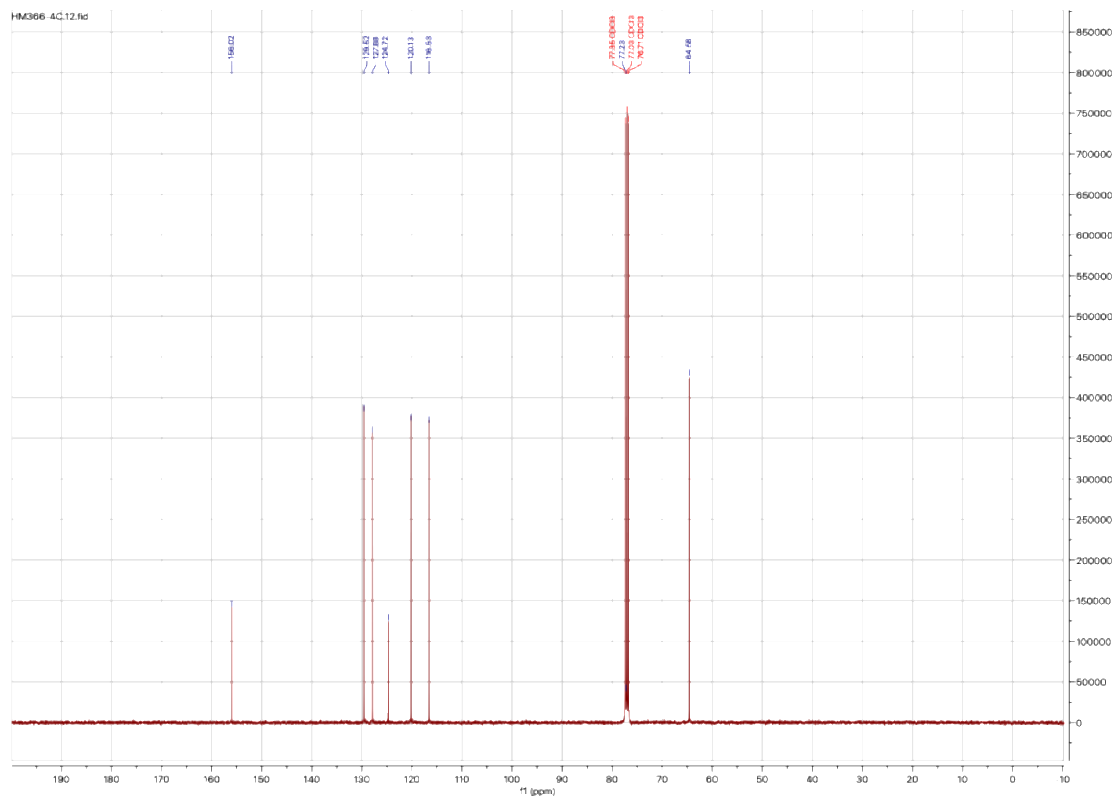
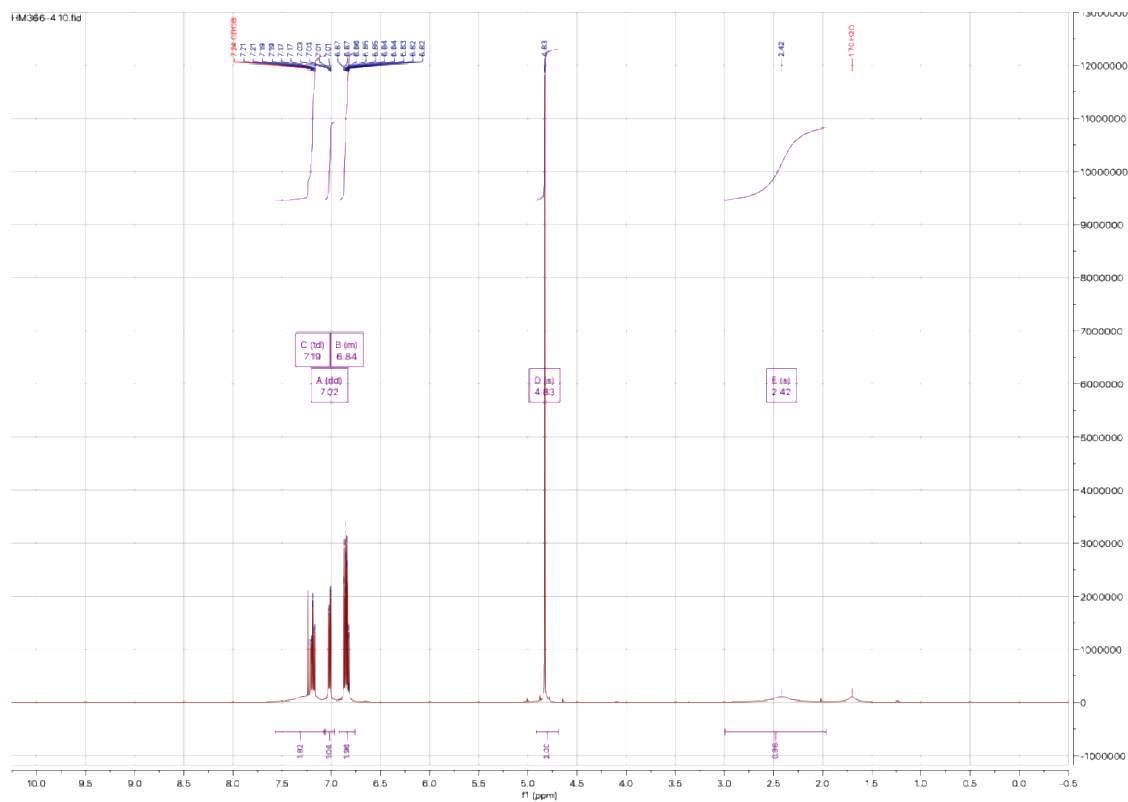
**2s**



# 2-(Hydroxymethyl)phenol (2t)



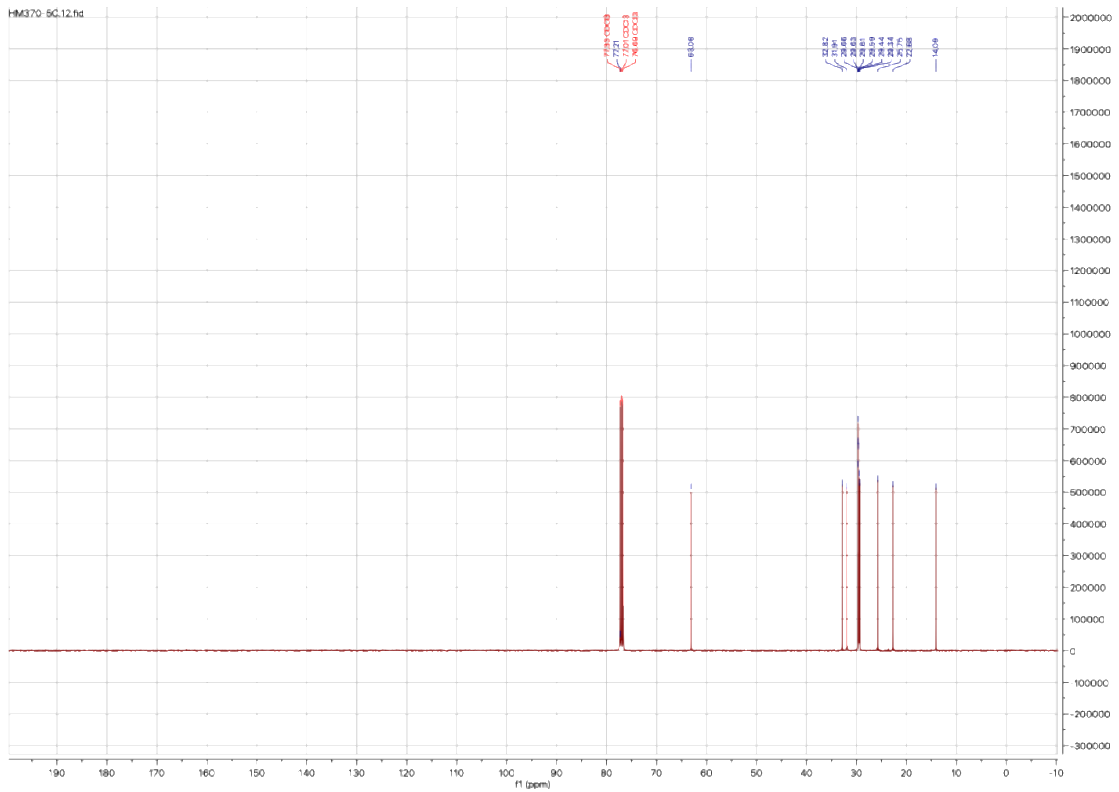
**2t**



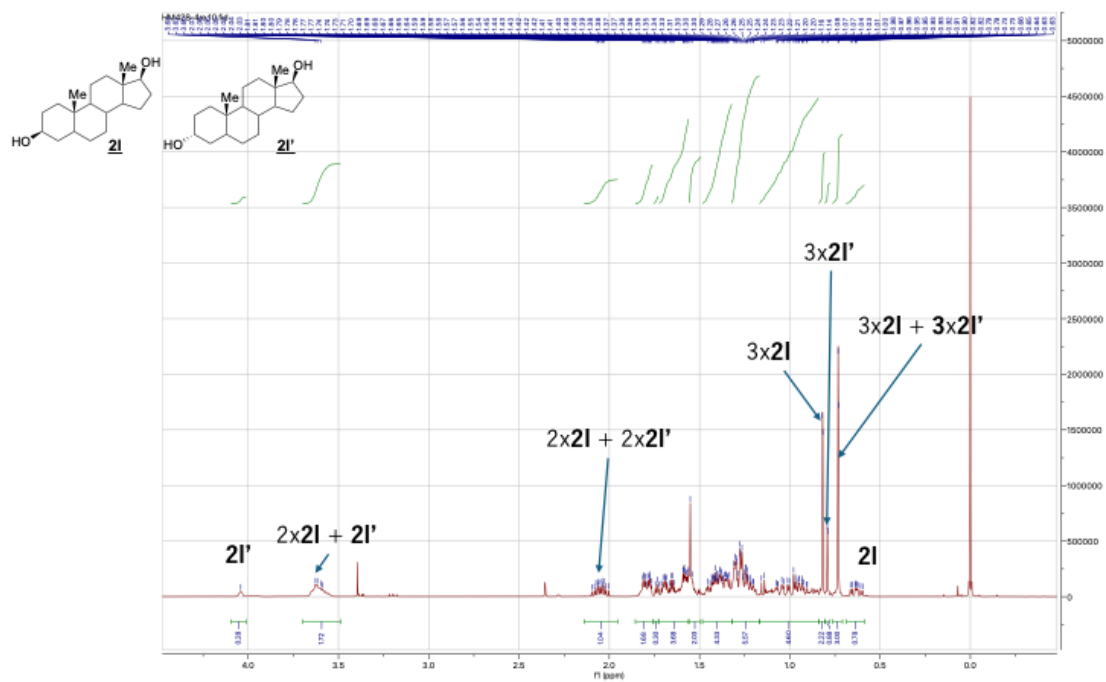
# 1-Dodecanol (2x)



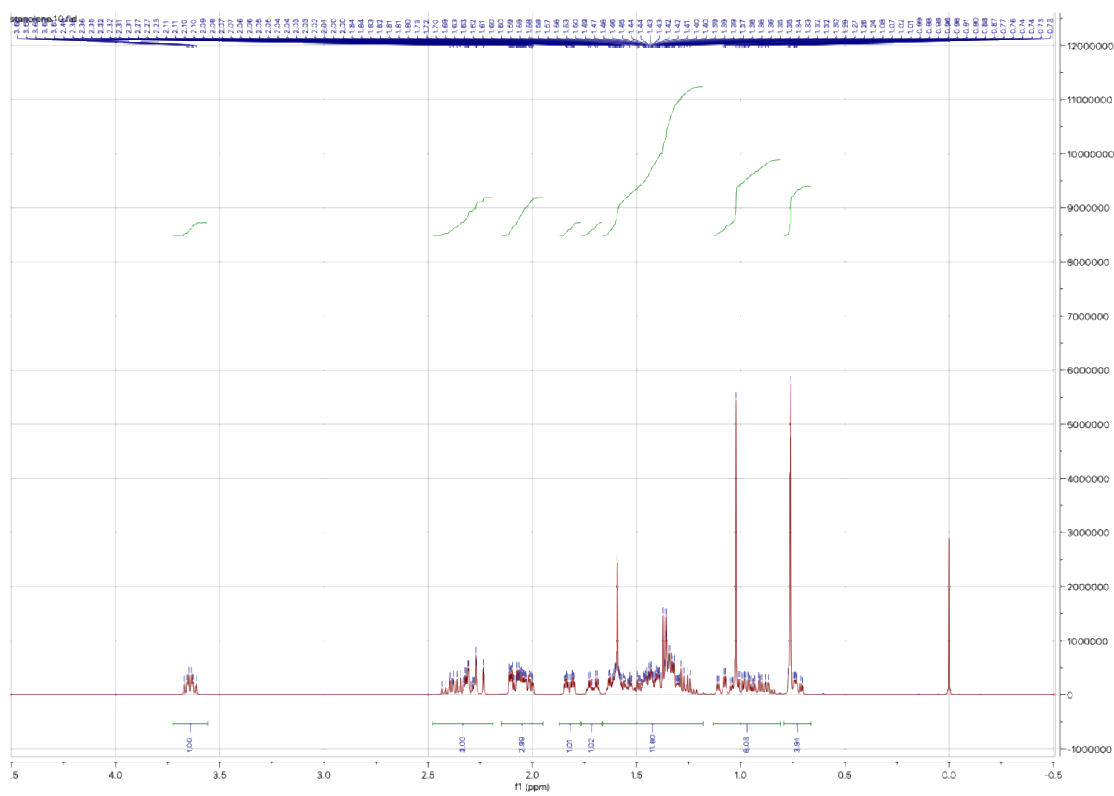
**2x**



2I + 2I' 2



<sup>1</sup>H NMR spectrum of stanolone (1I) (substrate)



## References

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