

Electronic supplementary information (ESI) for

**Supported Fe Catalysts Prepared through Layered Double
Hydroxides for Primary Amine Synthesis by Reductive Amination of
Carbonyl Compounds**

Yusuke Kita, Natsuki Yanagisawa, Mayu Arai, Keigo Kamata, Michikazu Hara

Laboratory for Materials and Structures, Institute of Innovative Research,
Tokyo Institute of Technology
Nagatsuta-cho 4259, Midori-ku, Yokohama 226-8503 (Japan)
Fax: (+81)45-924-5381
E-mail: hara.m.ae@m.titech.ac.jp

1. Materials
2. Instruments
3. Preparation of supported Fe catalysts by impregnation method
4. Procedure for IR measurements
5. Calculation method for surface coverage by carbon on supported Fe catalyst
6. Spectral data
7. NMR spectra
8. Additional tables and figures
9. References

1. Materials

The following materials were used as received: benzaldehyde (Aldrich), o-tolaldehyde (TCI), *p*-tolaldehyde (TCI), *p*-anisaldehyde (TCI), 4-fluorobenzaldehyde (TCI), 4-bromobenzaldehyde (Acros), furfural (TCI), 3-vinylbenzaldehyde (Aldrich), 2-heptanone (TCI), cyclohexanone (TCI), 2-adamantanone (TCI), acetophenone (TCI), benzonitrile (TCI), *trans*-cinnamaldehyde (TCI), 4-cyanostyrene (Wako), 4-nitrobenzaldehyde (Aldrich), cyclohexane (Kanto Chemical), toluene (Kanto Chemical), tetrahydrofuran (Wako), *N,N*-dimethylformamide (Kanto Chemical), ethanol (Kanto Chemical), dichloroethane (Kanto Chemical), isopropanol (Wako), CDCl_3 (Kanto Chemical), Fe_2O_3 (Aldrich), and acetylene carbon black (Wako). Raney alloy (Aldrich) was activated by the treatment of NaOH according to the report.^{S1}

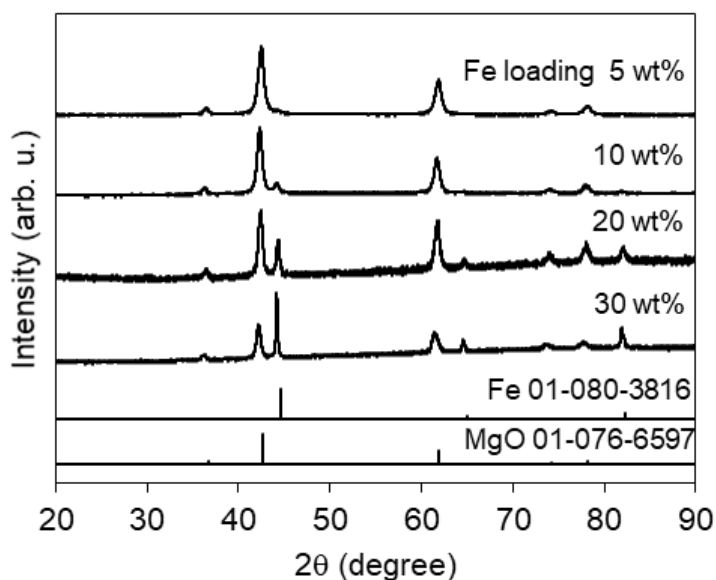
2. Instruments

Brunauer–Emmett–Teller (BET) specific surface areas of the samples were measured by nitrogen adsorption–desorption at $-196\text{ }^\circ\text{C}$ (Quantachrome Nova-4200e). Prior to the adsorption measurements, the samples were degassed in situ under vacuum at $150\text{ }^\circ\text{C}$ for 1 h. The BET surface area was determined using the multipoint BET algorithm in the P/P_0 range from 0.05 to 0.3. X-ray diffraction (XRD; Ultima IV, Rigaku) patterns of all samples were obtained using Cu $K\alpha$ radiation (40 kV, 40 mA) in the 2θ range of $5\text{--}90^\circ$. X-ray photoelectron spectroscopy (XPS; ESCA-3200 Shimadzu) measurements were performed using Mg $K\alpha$ radiation (1486.6 eV). Samples were fixed on double-sided carbon tape as a powder or a pellet. The binding energies were calibrated using sputtered C (1s peak at 285 eV) or vapor-deposited Au (84 eV). Raman spectroscopy was recorded by Raman spectrometer (NSRS-3100, JASCO) using a green laser at 532 nm. The slit width was 0.1 mm. CHN elemental analyses were conducted by Micro coder JM-10 (J-science Lab). Thermogravimetry-differential thermal analysis (TG-DTA) was performed using Thermo plus EV02 with smart loader (RIGAKU). The morphology of LDHs was characterized by field emission scanning electron microscope (FE-SEM) measurements were conducted using field emission scanning microanalyzer (JSM-6700FT, JEOL, Japan) at an accelerating voltage of 5 kV. Transmission electron microscopy (TEM; JEM-2100F, JEOL)-energy dispersive X-ray spectroscopy (EDS, JED-2300T, JEOL) measurements were conducted at an acceleration voltage of 200 kV. Gas-titration and temperature programmed reaction/desorption experiments were performed using an automatic measurement instrument (BELCAT-A, BEL Japan) with thermal conductivity (TCD) and mass (Belmass) detectors. For H_2 -pulse titration, the cell was loaded with catalyst in a glove box and placed in CATCryo without exposure to atmosphere. For pretreatment, hydrogen reduction was performed at $650\text{ }^\circ\text{C}$ for 20 minutes, and while the temperature was maintained at $650\text{ }^\circ\text{C}$, Ar substitution was performed to remove adsorbed hydrogen. Thereafter, the cell was transferred to a cryostat and cooled to $-60\text{ }^\circ\text{C}$ under an Ar flow, and pulse measurements were performed while maintaining the cell at $-60\text{ }^\circ\text{C}$ (carrier gas: Ar, adsorption gas: 5% H_2/Ar). Before the measurement of H_2 TPR for the mixture of Fe_2O_3 and carbon, 70 mg of Fe_2O_3 and 10 mg of carbon was mixed in a mortar for 20 minutes. ^1H NMR (400 MHz), and $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz)

spectra were measured on Bruker Avance III-400 spectrometers. All ^1H NMR chemical shifts were recorded in ppm (δ) relative to tetramethylsilane or referenced to the chemical shifts of residual solvent resonances (CHCl_3 was used as internal standard, δ 7.26). All ^{13}C NMR chemical shifts were recorded in ppm (δ) relative to carbon resonances in CDCl_3 at δ 77.16. The net amounts of metals were measured using inductively coupled plasma atomic emission spectroscopy (ICP-AES). The 30 mg of sample was calcined at 500 °C for 4 h and totally dissolved by HNO_3 . Gas chromatography (GC) analyses were conducted using a gas chromatography (GC-18A, Shimadzu) equipped with an InertCap 17 capillary column (internal diameter = 0.25 mm, length = 30 m) and a flame ionization detector (FID).

3. Preparation of supported Fe catalysts by impregnation method

20 wt% Fe/support_im: $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (1.013 g, 1.25 mmol), support (0.8 g) and water (50 mL) were added to the flask. After the evaporation, the catalyst was dried *in vacuo* at 100 °C for 1 h, calcined with N_2 flow at 400 °C for 2 h and reduced at 700 °C with H_2 flow for 2 h. The reduced catalyst was transferred into Ar-filled glovebox without exposure to air.



XRD patterns for Fe/MgO_im with different Fe loading.

4. Procedure for IR measurements

H_2 -probed IR measurements were conducted using scratched CaF_2 plate in an IR cell attached to a closed glass-circulation system. Fe catalyst was deposited on the scratched CaF_2 plate in Ar-filled glovebox. The sample was reduced with H_2 flow (80 mL/min) at 500 °C for 0.5 h. After replacing Ar in the closed glass-circulation system and the IR cell to H_2 by flowing H_2 (80 mL/min), the temperature was gradually decreased to 25 °C with measuring spectra.

D_2 -probed IR measurements were conducted using the same method as the H_2 probed measurement method, replacing H_2 with D_2 .

5. Calculation method for surface coverage by carbon on supported Fe catalyst

In the catalyst prepared by H₂ reduction of LDH-citrate at 600 °C, a peak due to metallic iron can be observed in XRD patterns, indicating that iron particle growth begins at around 550 °C. Calculations were made assuming that all carbon in the precursor remains at the temperature at which particle growth occurs. Since we do not know the structure of the carbon species, we laid out a single layer of carbon atoms on a two-dimensional surface with graphene structure and calculated the occupied area when viewed from above. The area may be smaller since the carbon species are thought to be layered.

Carbon content in the precursor for Fe-C/MgO (n): 2.98 wt%

Specific surface area of Fe-C/MgO (S_{BET}): 100 m² g⁻¹

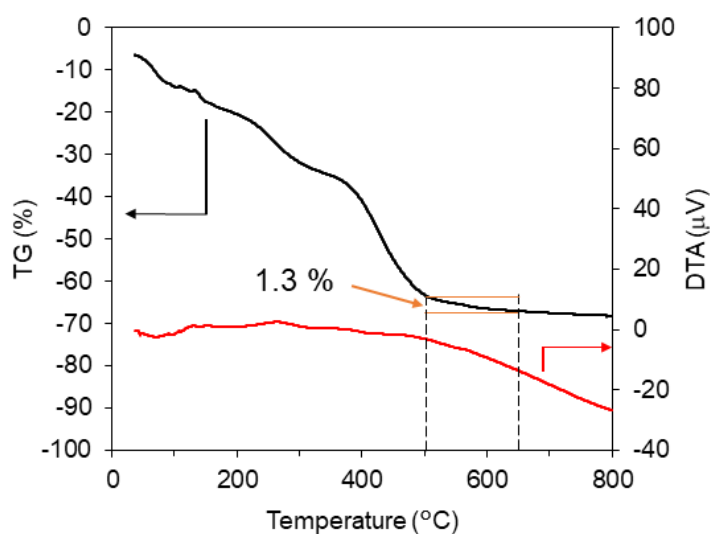
Area of graphene unit cell including two carbon (S_g): 5.34 × 10⁻²⁰ m²

Avogadro number (NA): 6.022 × 10²³ mol⁻¹

Molar mass (M_c): 12.011 g mol⁻¹

Surface coverage by carbon on Fe-C/MgO [%]

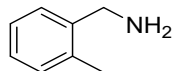
$$= \frac{n}{100} \times \frac{S_g \times NA}{M_c \times 2 \times S_{BET}} \times 100$$



TG-DTA profile with H₂ flow for LDH-citrate_cal

6. Spectral Data

o-Methylbenzylamine^{S2}

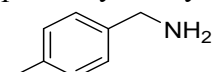


After the reaction was completed, the catalyst was separated by filtration.

¹H NMR (400 MHz, CDCl₃): δ = 7.31–7.15 (m, 4H), 3.86 (s, 2H), 2.34 (s, 3H), 1.74 (br, 2H).

¹³C NMR (100 MHz, CDCl₃): δ = 141.1, 135.6, 130.4, 127.2, 127.0, 126.3, 44.2, 18.9.

p-Methylbenzylamine^{S3}

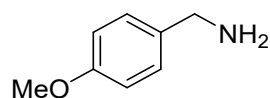


After the reaction was completed, the catalyst was separated by filtration.

¹H NMR (400 MHz, CDCl₃): δ = 7.18 (d, *J* = 7.9 Hz, 2H), 7.14 (d, *J* = 7.8 Hz, 2H), 3.81 (s, 2H), 2.33 (s, 3H), 1.64 (br, 2H).

¹³C NMR (100 MHz, CDCl₃): δ = 140.4, 136.5, 129.3, 127.2, 46.3, 21.2.

p-Methoxybenzylamine^{S3}

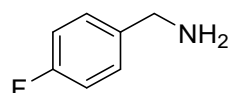


After the reaction was completed, the catalyst was separated by filtration.

¹H NMR (400 MHz, CDCl₃): δ = 7.26–7.21 (m, 2H), 6.87 (d, *J* = 8.4 Hz, 2H), 3.80 (s, 5H), 3.65 (s, 2H), 1.56 (br, 2H).

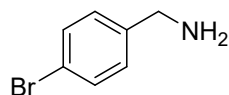
¹³C NMR (100 MHz, CDCl₃): δ = 158.7, 135.7, 128.4, 114.1, 55.4, 46.0.

4-Fluorobenzylamine



After the reaction, chlorobenzene was added to the reaction mixture as an external standard, and analyzed by GC measurement. Formation of the title compound was determined by GC-MS measurement.

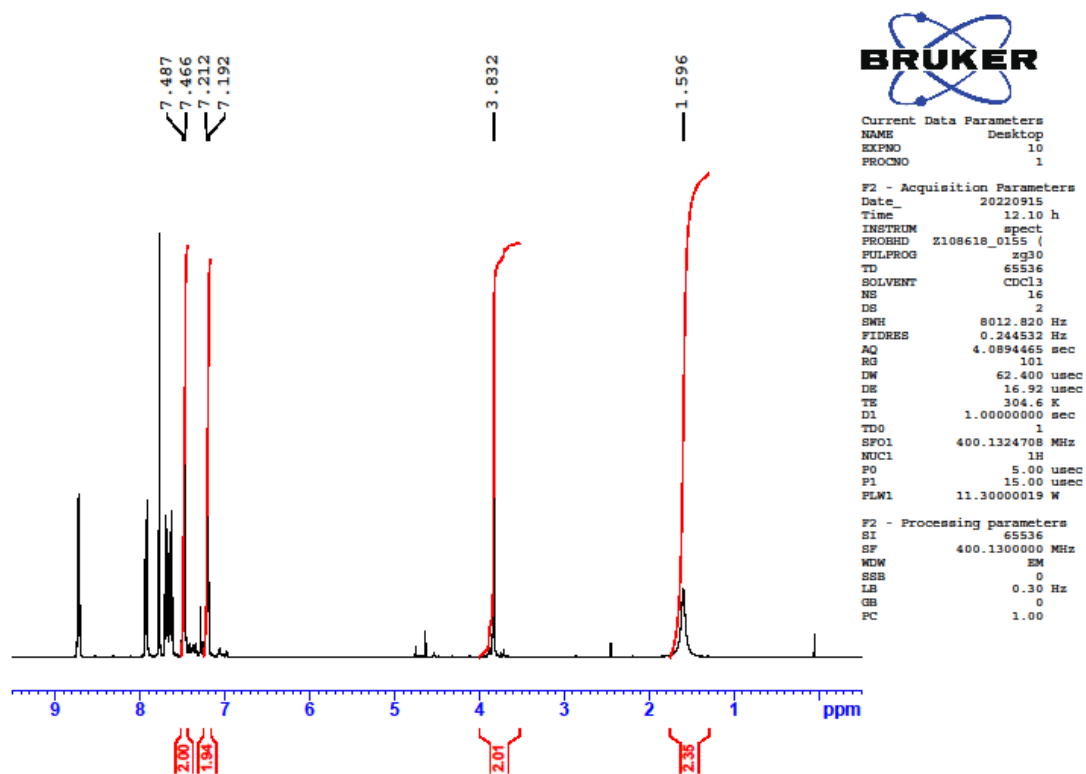
4-Bromobenzylamine^{S3}



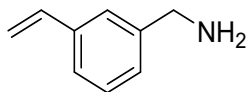
After the reaction, the catalyst was separated by filtration. The crude product was obtained by evaporation and analyzed by NMR analysis with phenanthrene as an internal standard. Formation of the title compound was determined by GC-MS measurement.

¹H NMR (400 MHz, CDCl₃): δ = 7.47 (d, *J* = 8.3 Hz, 2H), 7.20 (d, *J* = 8.2 Hz, 2H),

3.83 (s, 2H), 1.60 (br, 2H)

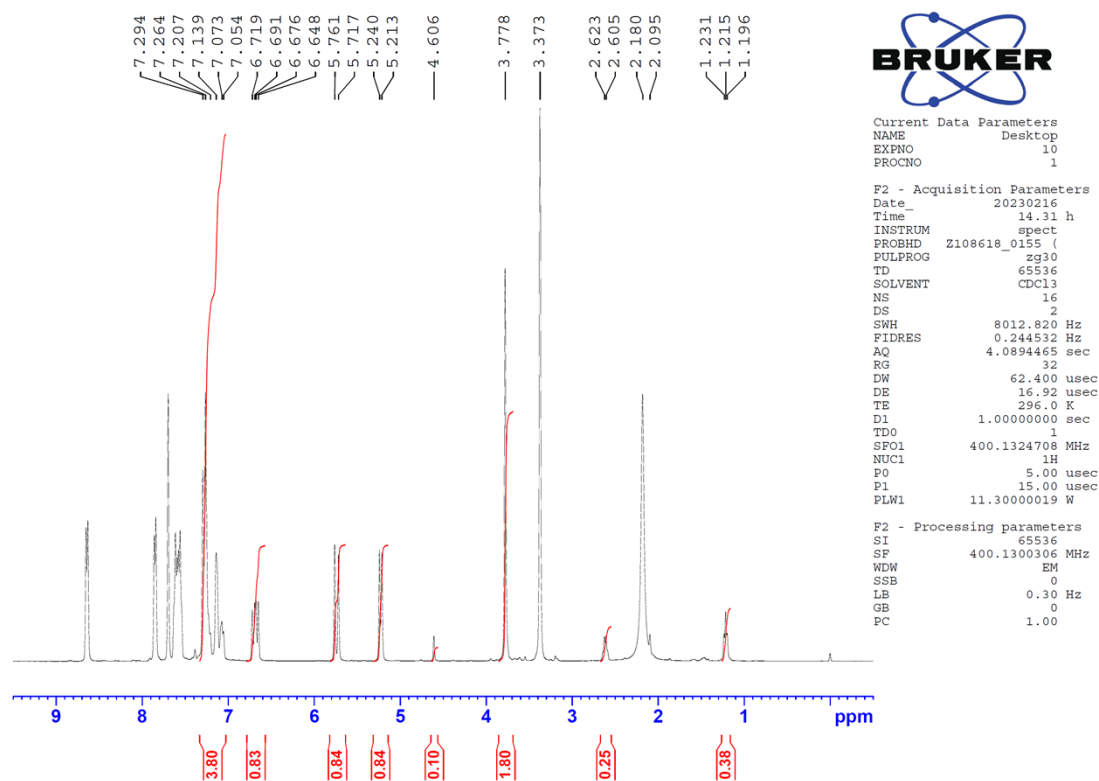


3-Vinylbenzylamine^{S3}

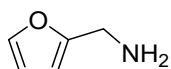


After the reaction, the catalyst was separated by filtration. The crude product was obtained by evaporation and analyzed by NMR analysis with phenanthrene as an internal standard. Formation of the title compound was determined by GC-MS measurement.

¹H NMR (400 MHz, CDCl₃): δ = 7.29–7.05 (m, 4H), 6.68 (dd, J = 11.2, 17.2 Hz, 1H), 5.74 (d, J = 17.6 Hz, 1H), 5.23 (d, J = 10.8 Hz, 1H), 3.78 (s, 2H).

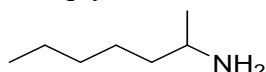


Furfurylamine



After the reaction, chlorobenzene was added to the reaction mixture as an external standard, and analyzed by GC measurement. Formation of the title compound was determined by GC-MS measurement.

2-Heptylamine^{S5}

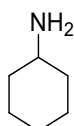


After the reaction was completed, the catalyst was separated by filtration.

¹H NMR (400 MHz, CDCl₃): δ = 2.87 (m, 1H), 1.30–1.16 (m, 8H), 1.05 (d, *J* = 4.7 Hz, 3H), 0.89 (t, *J* = 6.3 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃): δ = 47.1, 40.3, 32.1, 26.2, 24.1, 22.8, 14.1.

Cyclohexylamine^{S6}

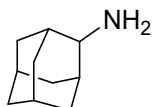


After the reaction was completed, the catalyst was separated by filtration.

¹H NMR (400 MHz, CDCl₃): δ = 2.66–2.58 (m, 1H), 1.83–1.80 (m, 2H), 1.74–1.69 (m, 2H), 1.62–1.53 (m, 3H), 1.31–1.21 (m, 2H), 1.17–0.99 (m, 3H).

^{13}C NMR (100 MHz, CDCl_3): $\delta = 50.6, 37.0, 25.8, 25.3$.

2-Aminoadamantane^{S7}

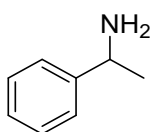


After the reaction was completed, the catalyst was separated by filtration.

^1H NMR (400 MHz, CDCl_3): $\delta = 2.98$ (s, 1H), 1.98 (d, $J = 12.6$ Hz, 2H), 1.86–1.72 (m, 10H), 1.53 (d, $J = 17.8$ Hz, 2H).

^{13}C NMR (100 MHz, CDCl_3): $\delta = 55.5, 38.0, 37.8, 35.2, 30.8, 27.8, 27.4$.

1-Phenylethylamine^{S8}

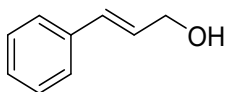


After the reaction was completed, the catalyst was separated by filtration.

^1H NMR (400 MHz, CDCl_3): $\delta = 7.32$ (m, 4H), 7.23 (m, 1H), 4.09 (q, $J = 6.3$ Hz, 1H), 1.65 (br s, 2H), 1.37 (d, $J = 6.3$ Hz, 3H)

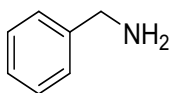
^{13}C NMR (100 MHz, CDCl_3): $\delta = 147.8, 128.5, 126.9, 125.7, 51.4, 25.7$.

Cinnamyl alcohol



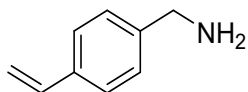
After the reaction, chlorobenzene was added to the reaction mixture as an external standard, and analyzed by GC measurement. Formation of the title compound was determined by GC-MS measurement.

Benzylamine



After the reaction, chlorobenzene was added to the reaction mixture as an external standard, and analyzed by GC measurement. Formation of the title compound was determined by GC-MS measurement.

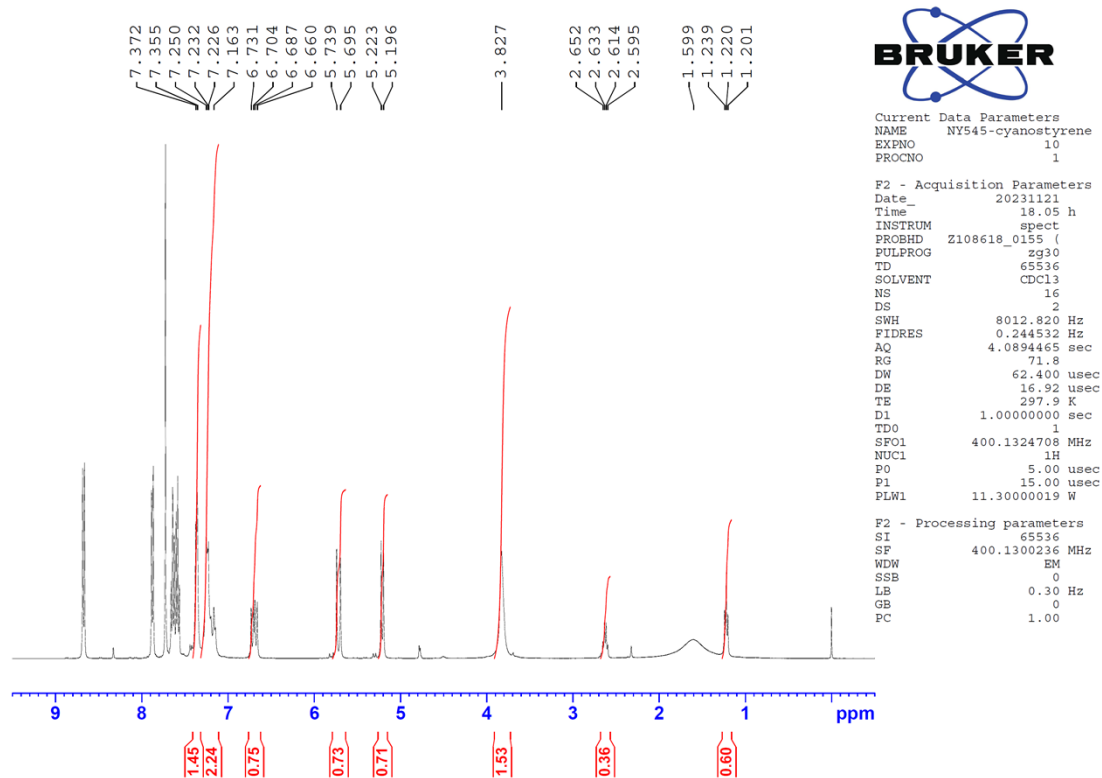
4-vinylbenzylamine



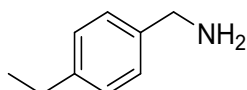
After the reaction, the catalyst was separated by filtration. The crude product was obtained by evaporation and analyzed by NMR analysis with phenanthrene as an internal standard. Formation of the title compound was determined by GC-MS

measurement.

^1H NMR (400 MHz, CDCl_3): $\delta = 7.36$ (d, $J = 6.8$ Hz, 2H), 7.24 (d, $J = 7.1$ Hz, 2H), 6.70 (dd, $J = 17.6, 10.8$ Hz, 1H), 5.72 (d, $J = 17.6$ Hz, 1H), 5.21 (d, $J = 10.8$ Hz, 1H), 3.83 (s, 2H).

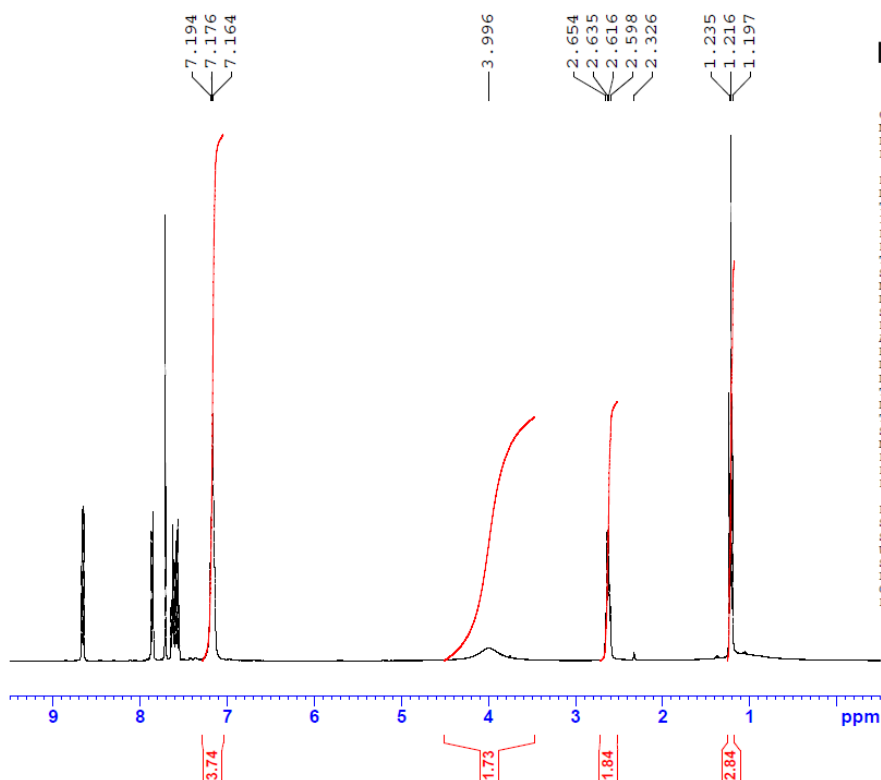


4-Ethylbenzylamine



After the hydrogenation of 4-vinylbenzocnitrile over Raney Ni, the catalyst was separated by filtration. The crude product was obtained by evaporation and analyzed by NMR analysis with phenanthrene as an internal standard. Formation of the title compound was determined by GC-MS measurement.

^1H NMR (400 MHz, CDCl_3): $\delta = 7.19$ – 7.16 (m, 4H), 4.00 (br s, 2H), 2.63 (q, $J = 7.5$ Hz, 2H), 1.22 (t, $J = 7.6$ Hz, 3H).



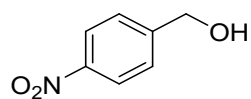
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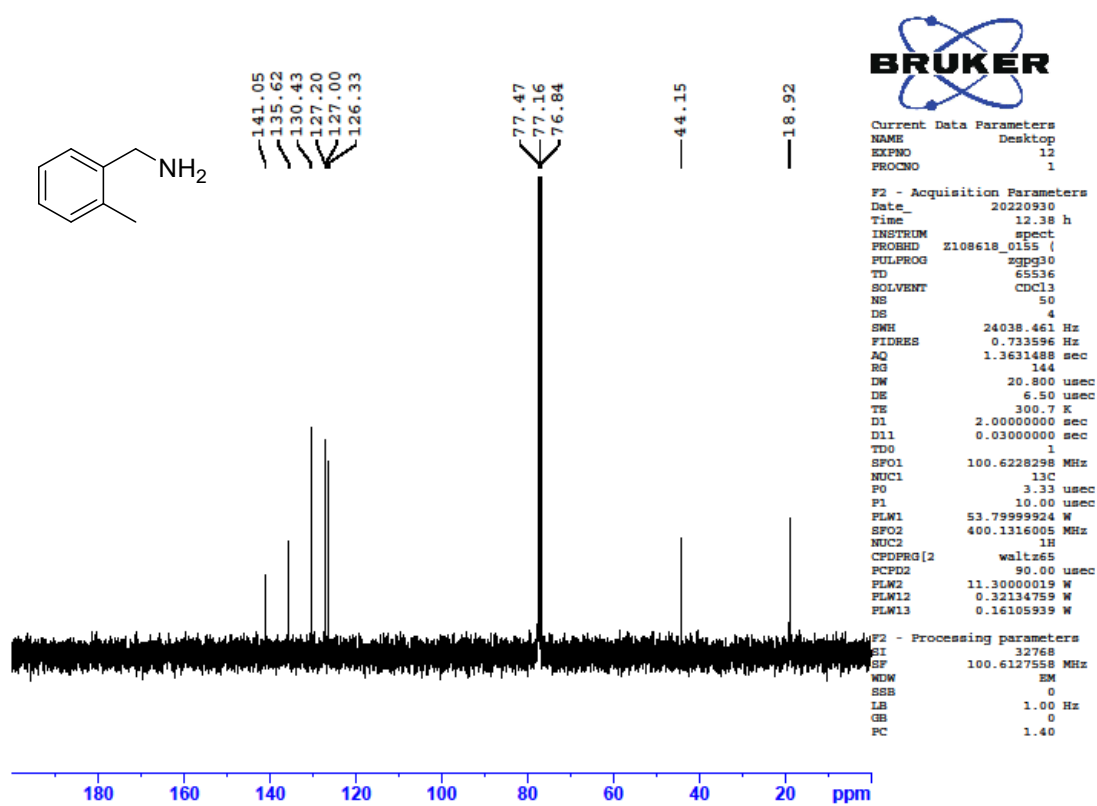
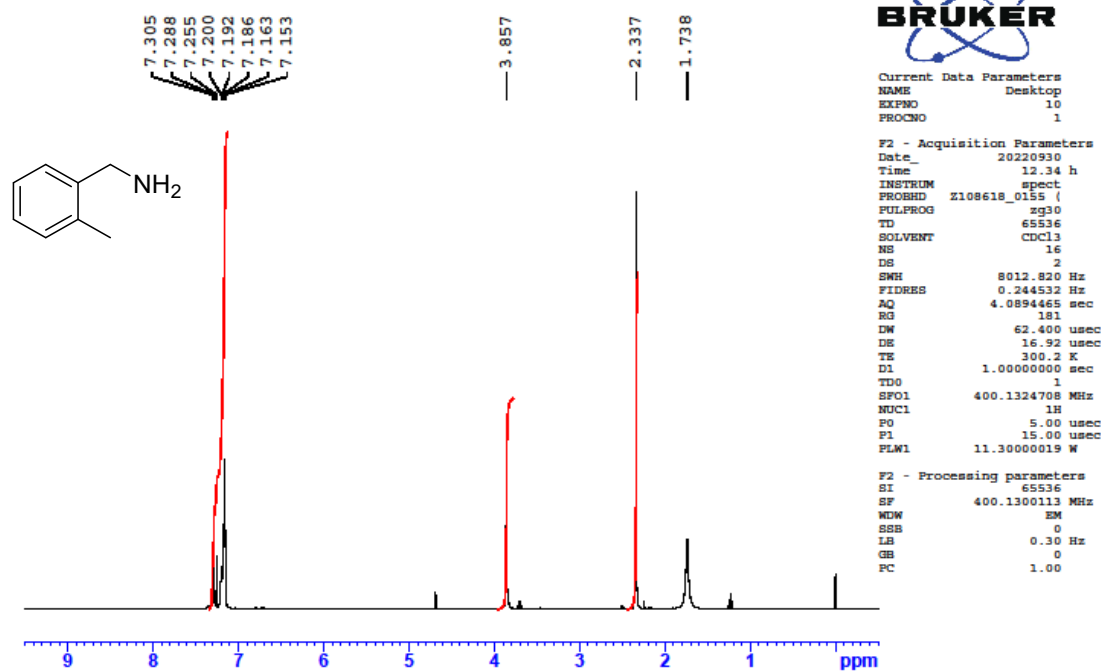
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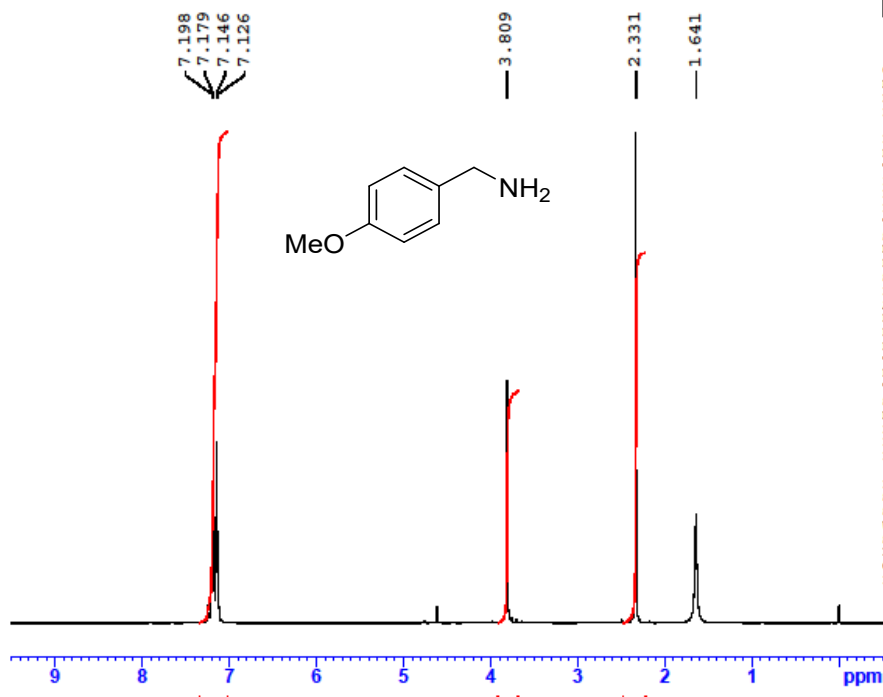
4-Nitrobenzyl alcohol



After the reaction, chlorobenzene was added to the reaction mixture as an external standard, and analyzed by GC measurement. Formation of the title compound was determined by GC-MS measurement.

7. NMR spectra

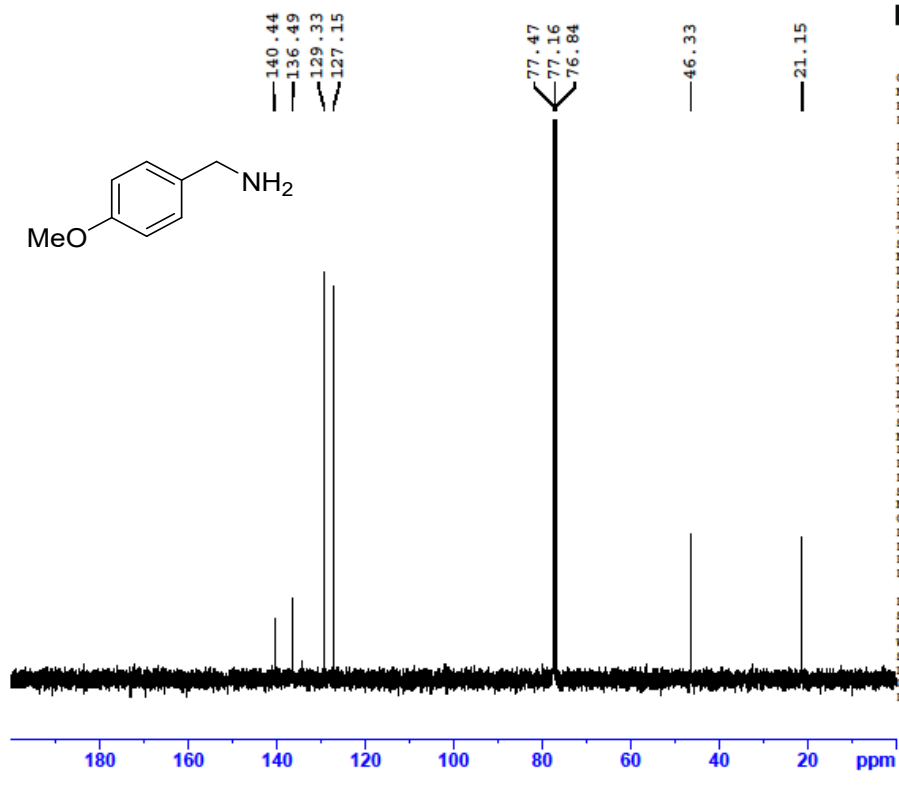




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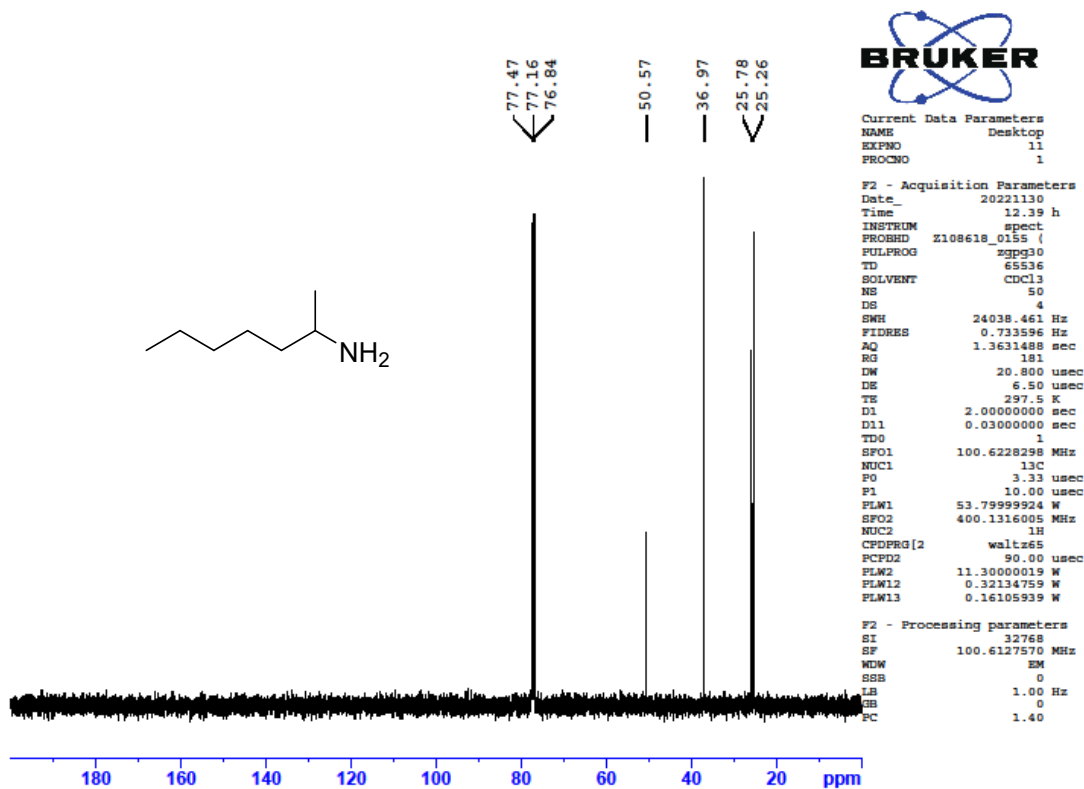
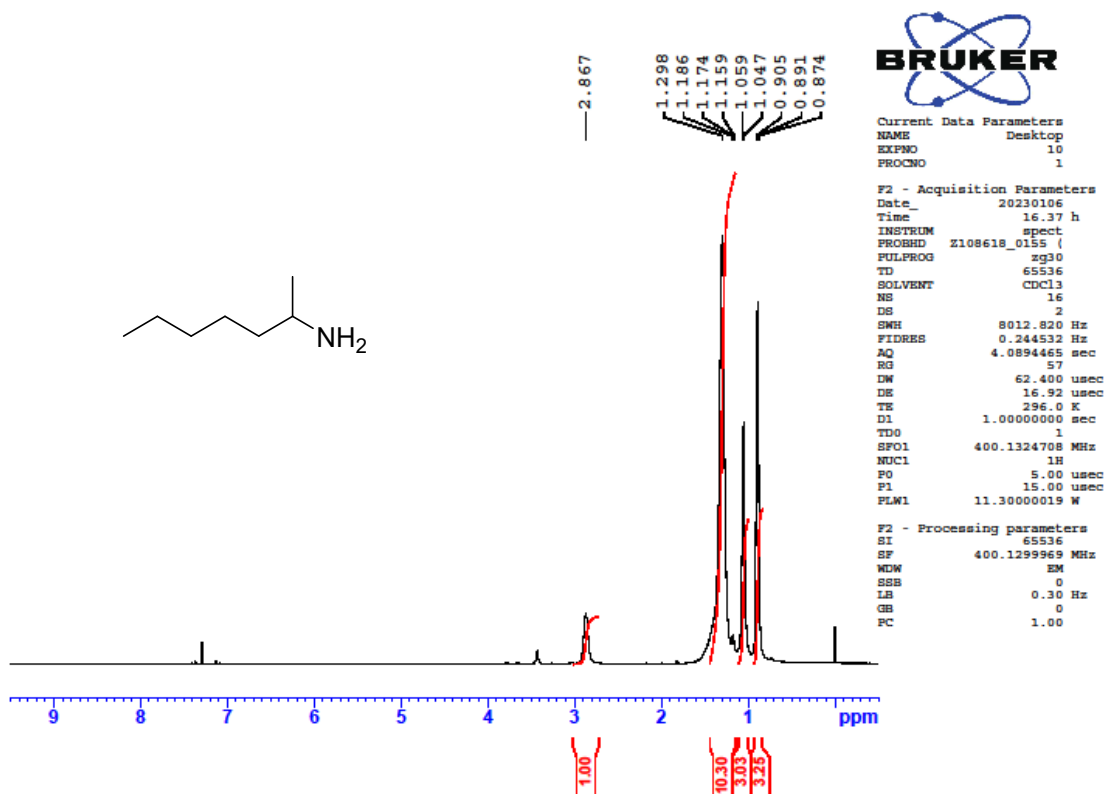
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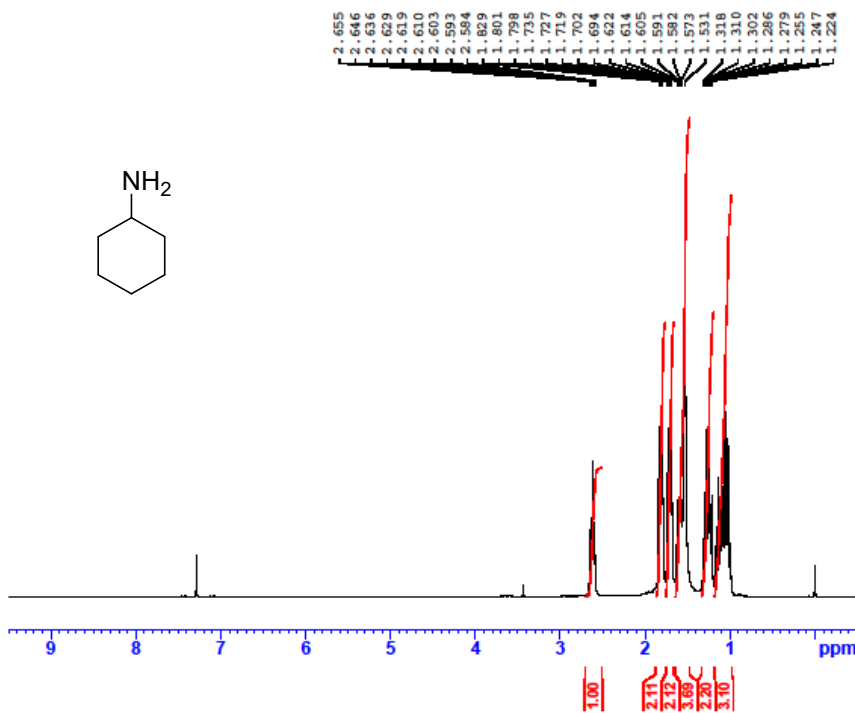


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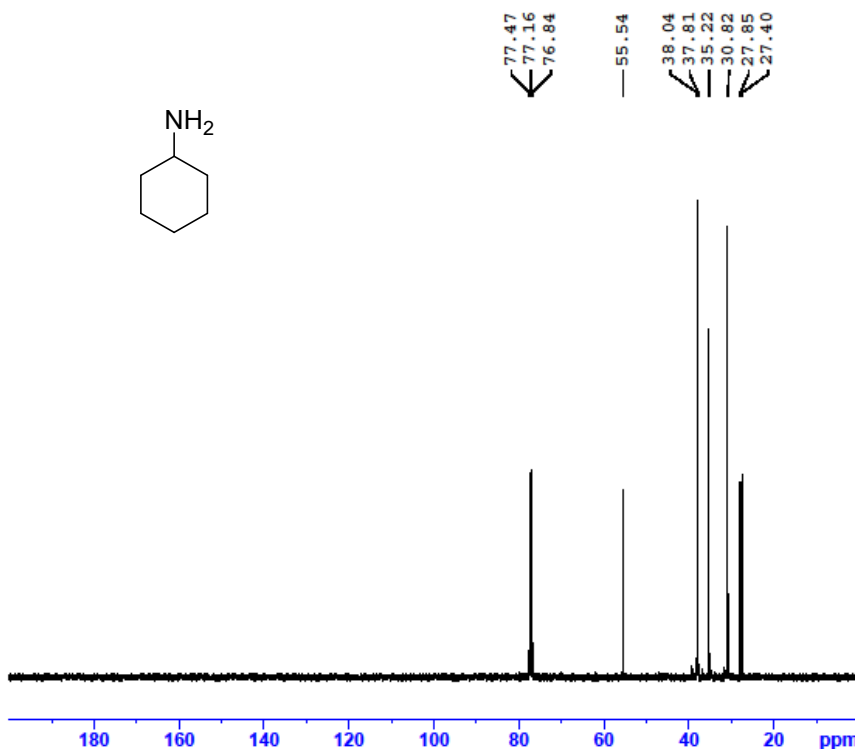




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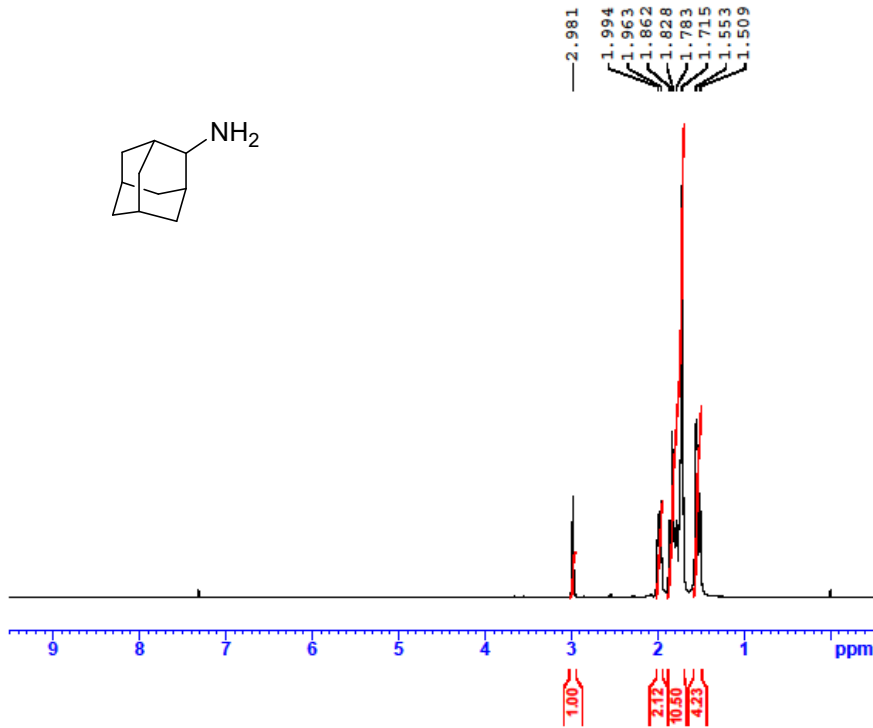
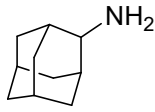
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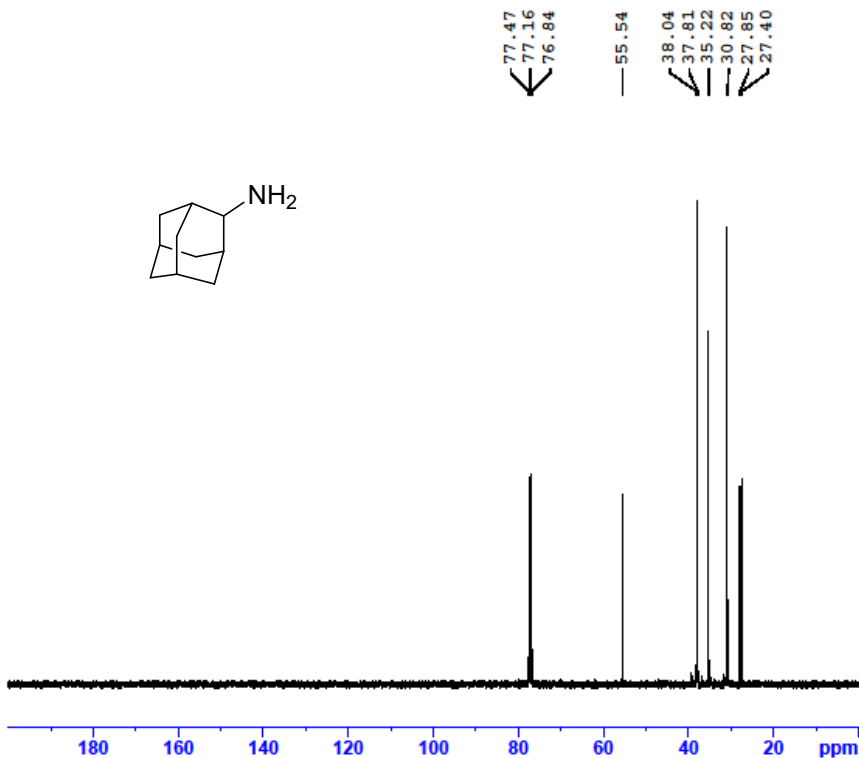
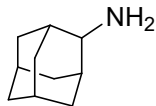
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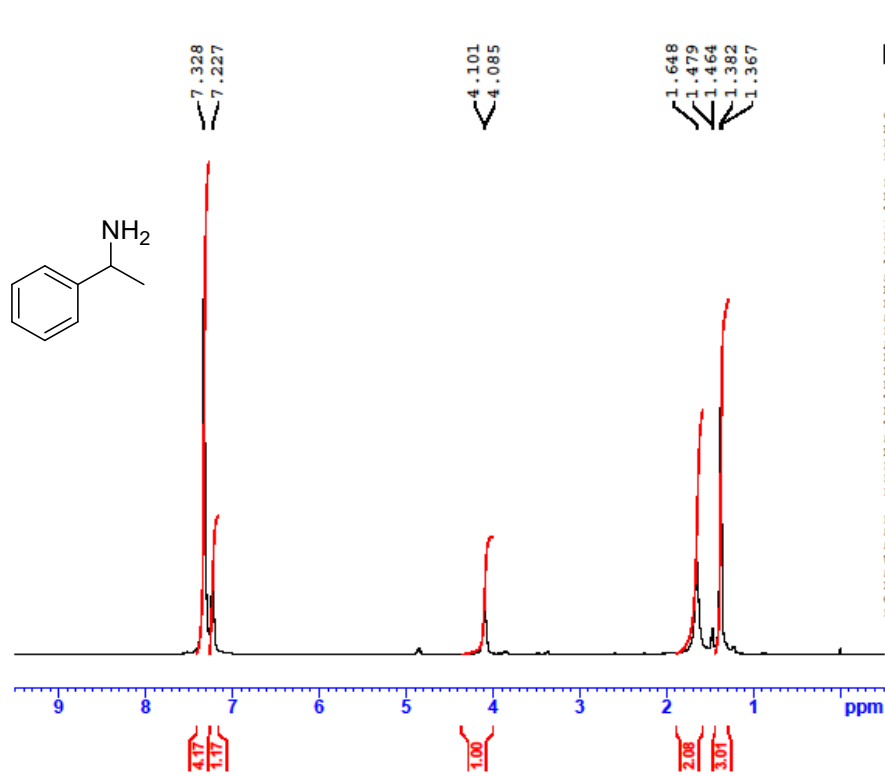
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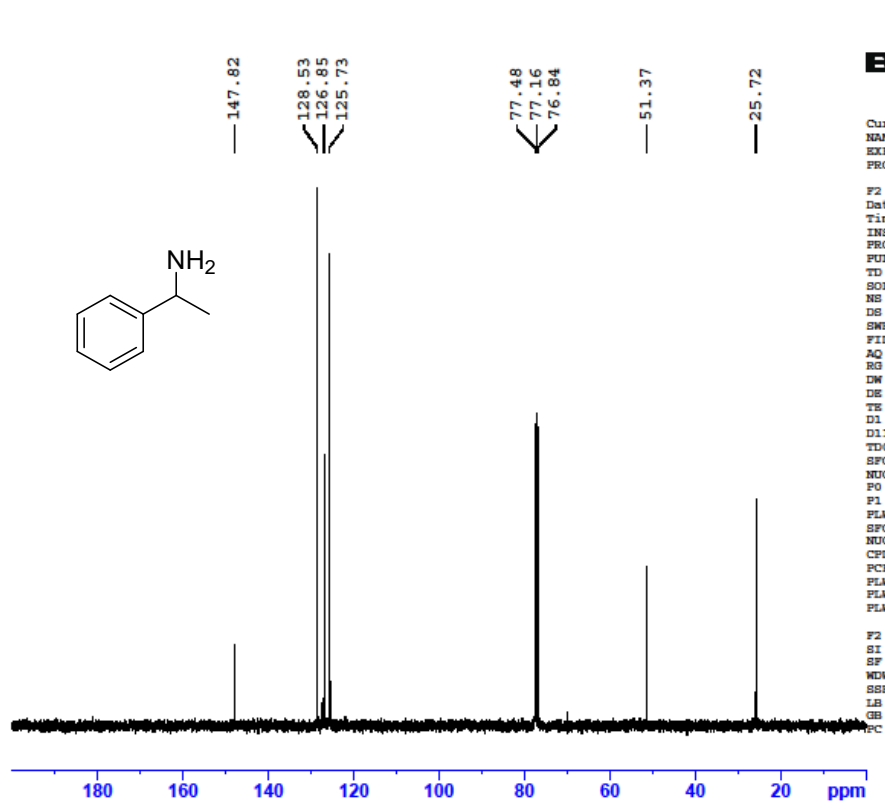
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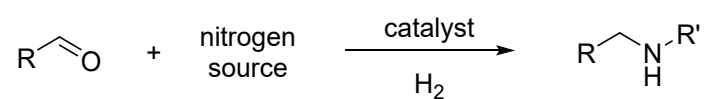
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8. Additional tables and figures

Table S1 Reported Fe catalysts for reductive amination using H₂



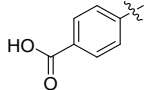
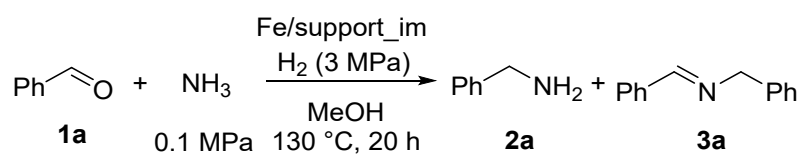
catalyst	Fe loading (mol%)	R	Nitrogen source	$p\text{H}_2$ (MPa)	Temp. (°C)	Reaction time (h)	Yield (%)	ref
Fe ₂ O ₃ /NGr @C	5	Phenyl	NH ₃ aq.	7	170	30	85	S9
Fe(N)SiC	10	Phenyl	PhNO ₂	6.5	130	20	89	S10
Fe-P ₉₀₀ -PCC	0.025		NH ₃ aq.	6	75	30	98	S11
Fe-P ₉₀₀ -PCC	0.025	Phenyl	MeNH ₂	6	120	4	98	S11
Fe@HC ₇₀₀	2.3	Phenyl	NH ₃ in MeOH	2	110	4	92	S12
Fe-C/MgO	12	Phenyl	NH ₃	1.5	80	20	99	This work

Table S2 Reductive amination of **1a** over Fe/support_im^a

Entry	Support	Crystallite size (nm)	S _{BET} (m ² g ⁻¹)	Conv. (%)	Yield (%)	
					2a	3a
1	SiO ₂	17	195	99	0	72
2	TiO ₂	43	19	92	2	32
3	Nb ₂ O ₅	34	-	>99	33	24
4	Al ₂ O ₃	22	178	94	1	26
5	ZrO ₂	34	37	>99	51	31
6	MgO	25	29	>99	56	28

^a Reaction conditions: Fe/support_im (0.02 g), **1a** (0.5 mmol), MeOH (5 mL), *p*NH₃ (0.1 MPa), *p*H₂ (3 MPa), 20 h. Conversion and yield were determined by GC analysis.

Table S3 Effects of reduction temperature over Fe-C/MgO^a

$$\begin{array}{c}
 \text{Ph}-\text{CHO} + \text{NH}_3 \xrightarrow[\text{MeOH}]{\text{Fe-C/MgO, H}_2 (1.5 \text{ MPa})} \text{Ph}-\text{CH}_2\text{NH}_2 + \text{Ph}-\text{CH}=\text{N}-\text{CH}_2\text{Ph} \\
 \text{1a} \quad 0.1 \text{ MPa} \quad 80 \text{ }^\circ\text{C, 20 h} \quad \text{2a} \quad \text{3a}
 \end{array}$$

Entry	Reduction temp. (°C)	S _{BET} (m ² /g)	Crystallite size (nm)		Conv. (%)	Yield (%)	
			Fe	MgO		2a	3a
1	650	100	7	9.5	>99	99	-
2 ^b	650	98	7	9.5	>99	88	3
2	600	119	-	7.8	>99	97	4
3	550	142	-	6.2	>99	59	24
4	500	168	-	5.5	96	-	17

^a Reaction conditions: Fe-C/MgO* (citric acid/Fe = 2.5, 0.02 g), **1a** (0.5 mmol), MeOH (5 mL), *p*NH₃ (0.1 MPa), *p*H₂ (1.5 MPa), 80 °C, 20 h. Conversion and yield were determined by GC analysis. ^b Catalyst was prepared by the reduction of MgFe-LDH in H₂ flow (30 mL/min). * Catalysts were prepared by the reduction of MgFe-LDH-citrate in H₂ flow (100 mL/min).

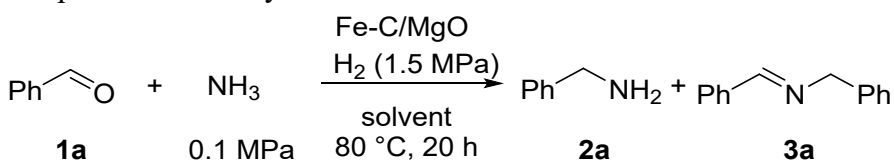
Table S4 Effects of Fe loading in Fe/MgO_{im} on reductive amination of **1a**^a

$$\begin{array}{c}
 \text{Ph}-\text{CHO} + \text{NH}_3 \xrightarrow[\text{solvent}]{\text{Fe/MgO}_{im}, \text{H}_2 (3 \text{ MPa})} \text{Ph}-\text{CH}_2\text{NH}_2 + \text{Ph}-\text{CH}=\text{N}-\text{CH}_2\text{Ph} \\
 \text{1a} \quad 0.1 \text{ MPa} \quad 130 \text{ }^\circ\text{C, 20 h} \quad \text{2a} \quad \text{3a}
 \end{array}$$

Entry	Catalyst			Conv. (%)	Yield (%)	
	Fe loading (wt%)	Crystallite Size (nm)	Loading amount (mg)		2a	3a
1	1	-	20	95	1	6
2	1	-	100	98	2	19
3	5	-	20	>99	85	<1
4	5	-	80	>99	79	<1
5	10	16	40	>99	86	1
6	20	21	20	>99	68	17
7	30	43	13	94	0	6

^a Reaction conditions: Fe/MgO-im, **1a** (0.5 mmol), MeOH (5 mL), *p*NH₃ (0.1 MPa), *p*H₂ (3 MPa), 130 °C, 20 h. Conversion and yield were determined by GC analysis

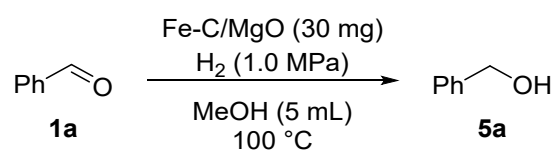
Table S5 Optimization study^a



Entry	Catalyst amount (g)	Temp (°C)	Solvent	H ₂ (MPa)	NH ₃ (MPa)	Conv . (%)	Yield (%)	
							2a	3a
1	0.02	60	MeOH	1.0	0.1	98	-	16
2	0.02	100	MeOH	1.5	0.1	>99	81	6
3 ^b	0.015	110	MeOH	2.0	0.1	>99	88	8
4	0.02	80	EtOH	1.5	0.1	>99	50	22
5	0.02	80	DMF	1.5	0.1	46	1	20
6	0.02	80	THF	1.5	0.1	63	1	12
7	0.02	80	cyclohexane	1.5	0.1	85	5	9
8	0.02	80	toluene	1.5	0.1	73	2	6
9	0.02	80	dichloroethane	1.5	0.1	81	-	trace
10	0.02	80	MeOH	1.5	0.3	>99	46	7
11	0.02	80	MeOH	2.0	0.3	>99	91	0
12	0.04	80	MeOH	1.5	0.1	>99	88	1
13	0.04	80	MeOH	1.0	0.1	>99	95	1
14	0.02	80	MeOH	2.0	0.1	>99	87	4

^a Reaction conditions: Fe-C/MgO (0.02 g), **1a** (0.5 mmol), solvent (5 mL), *p*NH₃ (x MPa), *p*H₂ (y MPa), 80 °C, 20 h. Conversion and yield were determined by GC analysis.

^b Run for 4 h.

Table S6 Comparison of Fe-C/MgO and Fe/MgO on hydrogenation of **1a**^a

Catalyst	Time (h)	Conv. (%)	Yield of 5a (%)
Fe-C/MgO	1	77	77
	8	10	3
Fe/MgO	70	86	79

^a Reaction conditions: catalyst (0.03 g), **1a** (0.5 mmol), MeOH (5 mL), $p\text{H}_2$ (1.0 MPa), 100 °C. Conversion and yield were determined by GC analysis.

Table S7 Surface and bulk composition of Fe and Mg

Catalyst	Precursor	Surface composition (Fe/Mg) ^a	Bulk composition (Fe/Mg) ^b
Fe/MgO	LDH-CO ₃	1.3 / 98.7	14.6 / 85.4
Fe-C/MgO	LDH-Citrate	1.5 / 98.5	14.9 / 85.1

^a Determined by XPS measurements. ^b Determined by ICP-AES measurements.

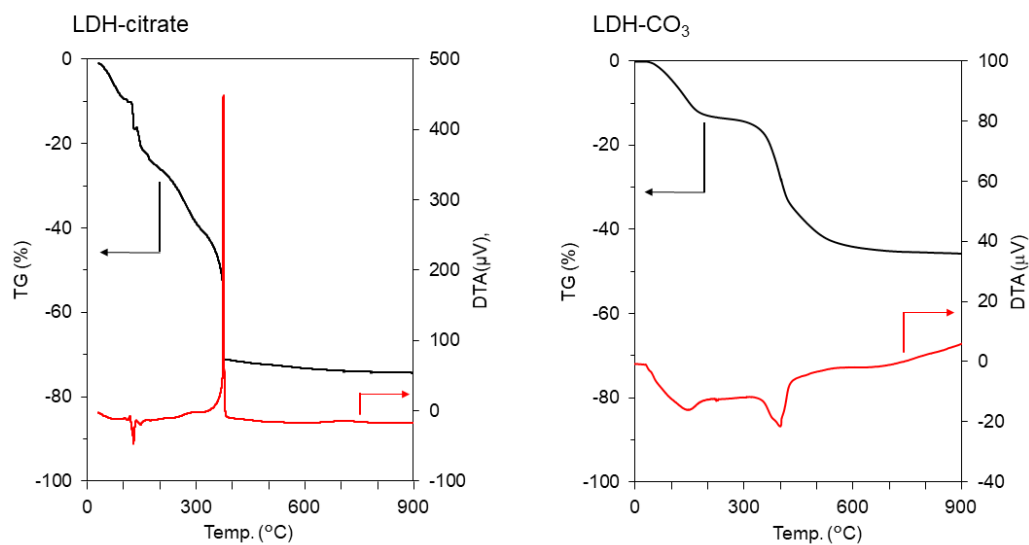


Fig. S1 TG-DTA analysis for LDH-CO₃ and LDH-citrate

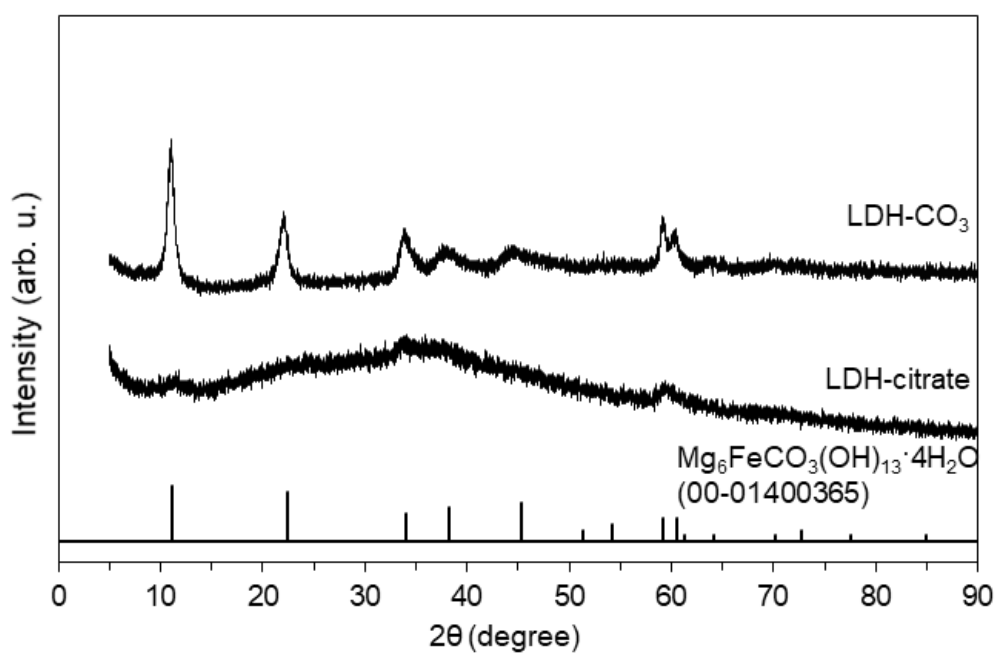


Fig. S2 XRD patterns for LDH-CO₃ and LDH-citrate.

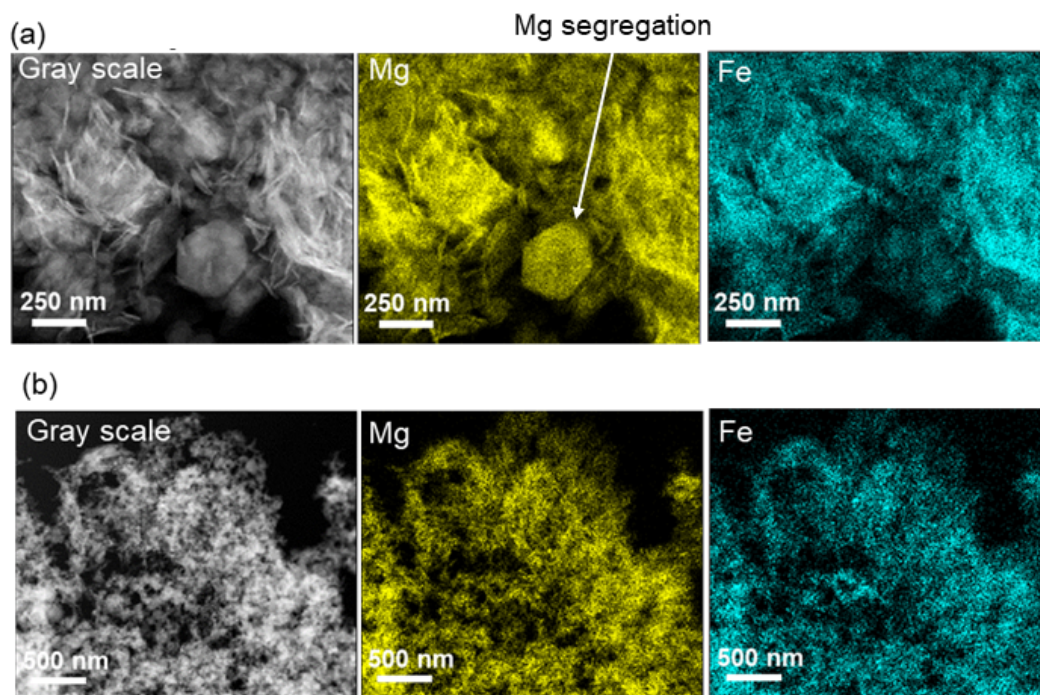


Fig. S3 TEM-EDS mapping for (a) MgFe-LDH-CO₃ and (b) MgFe-LDH-citrate.

Since the Mg segregation disappeared with the introduction of citric acid, Mg species such as Mg(OH)₂ can be once dissolved in the solution and re-precipitated to be incorporated into the LDH layer during the introduction of citrate.

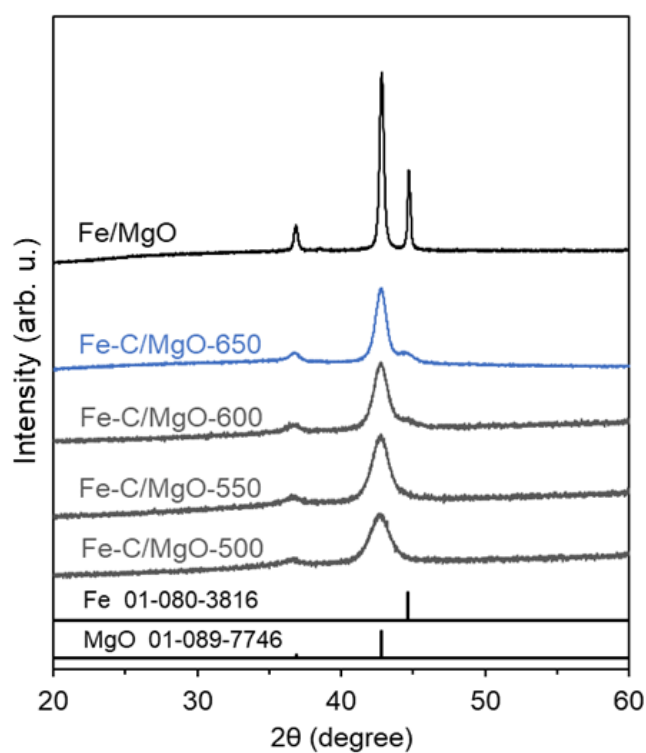


Fig. S4 XRD patterns for Fe/MgO with different reduction temperature.

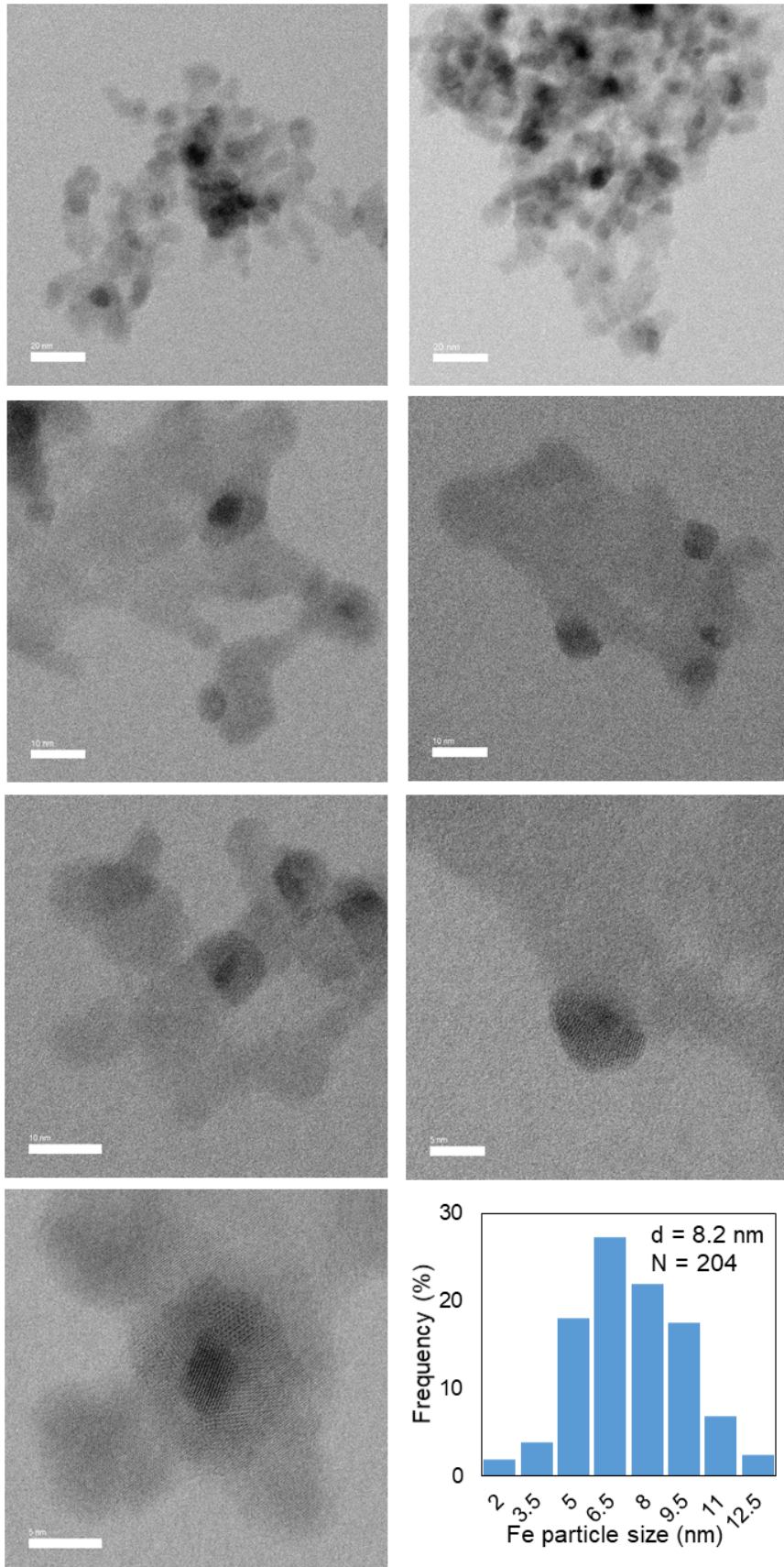


Fig. S5 STEM images of Fe-C/MgO

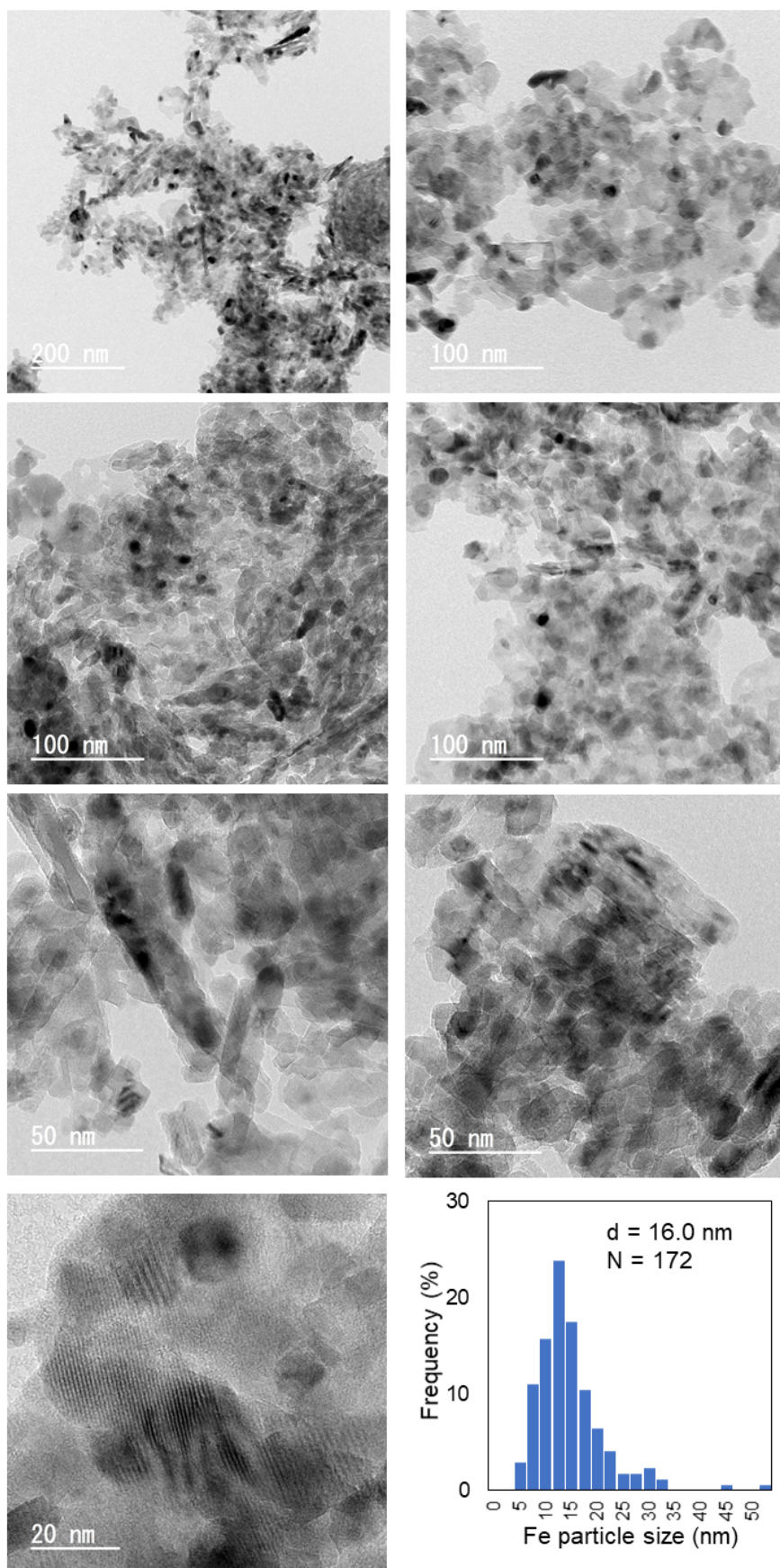
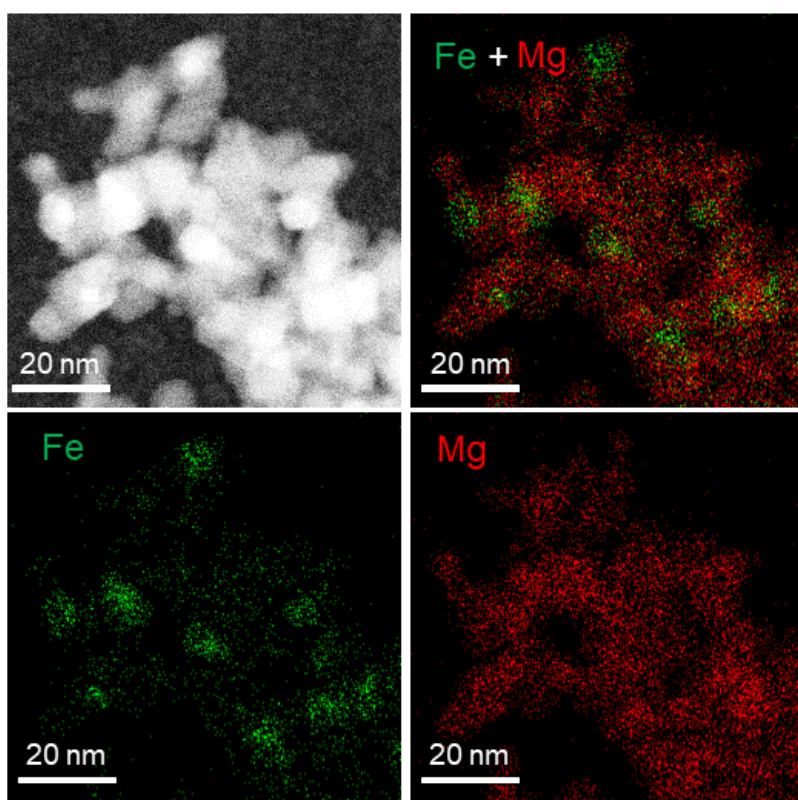


Fig. S6 STEM images of Fe/MgO



Entry		Atomic ratio Fe / Mg
1	area A	85 / 15
2	area B	53 / 47
3	area C	9 / 91
.....		
4	total	14.6 / 85.4 ^a

^a Determined by ICP measurement.

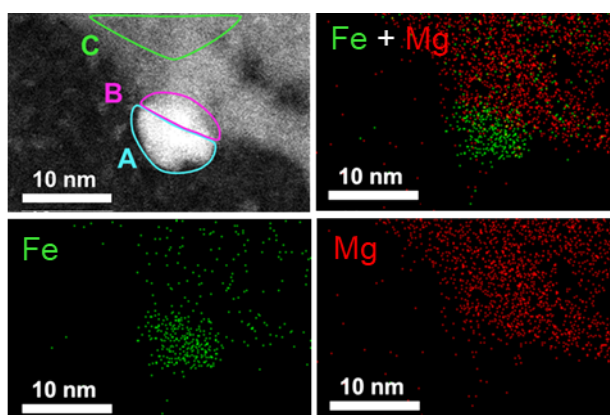


Fig. S7 TEM-EDS mapping for Fe-C/MgO

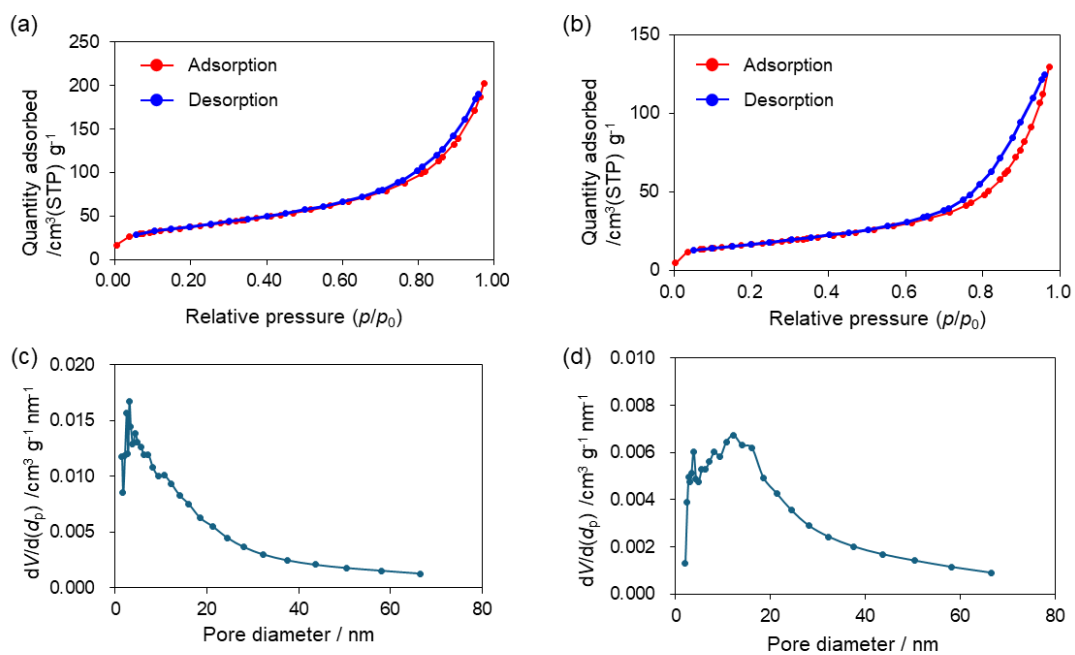
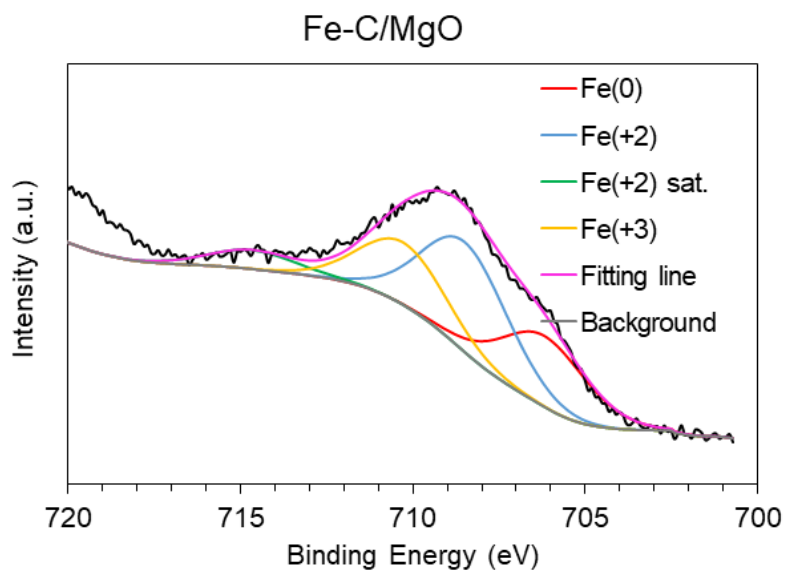
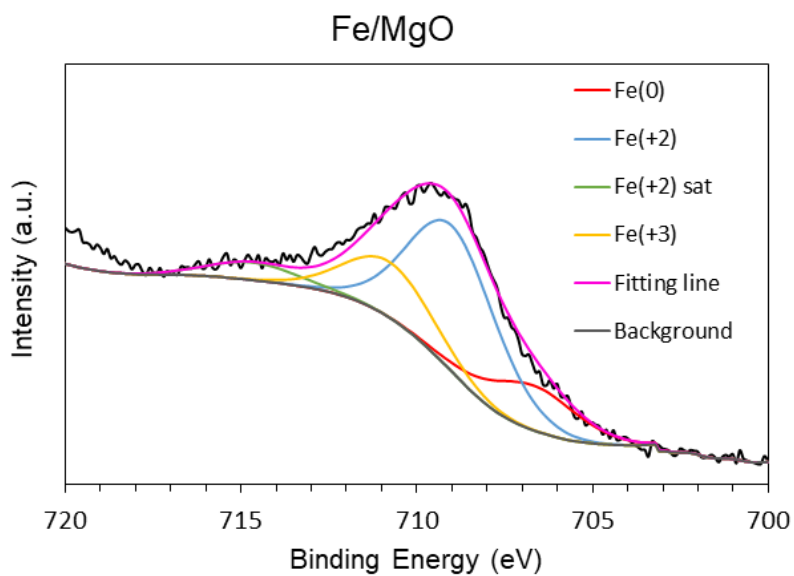


Fig. S8 N₂ sorption isotherm of (a) Fe-C/MgO and (b) Fe/MgO and BJH pore size distribution plot of (c) Fe-C/MgO and (d) Fe/MgO



Relative ratio (%)
 $\text{Fe}^0 : \text{Fe}^{2+} : \text{Fe}^{3+} = 29.1 : 45.1 : 25.7$



Relative ratio (%)
 $\text{Fe}^0 : \text{Fe}^{2+} : \text{Fe}^{3+} = 18.2 : 59.1 : 22.6$

Fig. S9 Deconvolution of Fe 2p XPS spectra for Fe-C/MgO and Fe/MgO

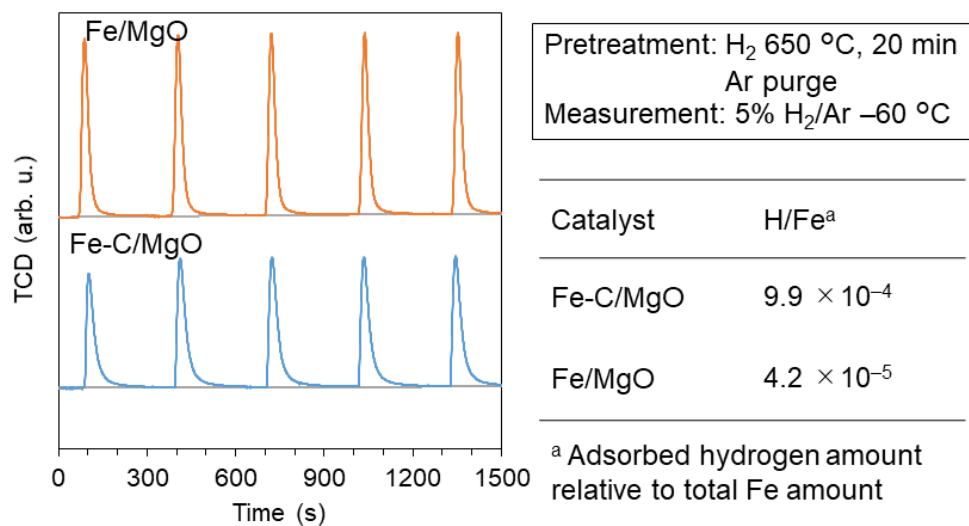
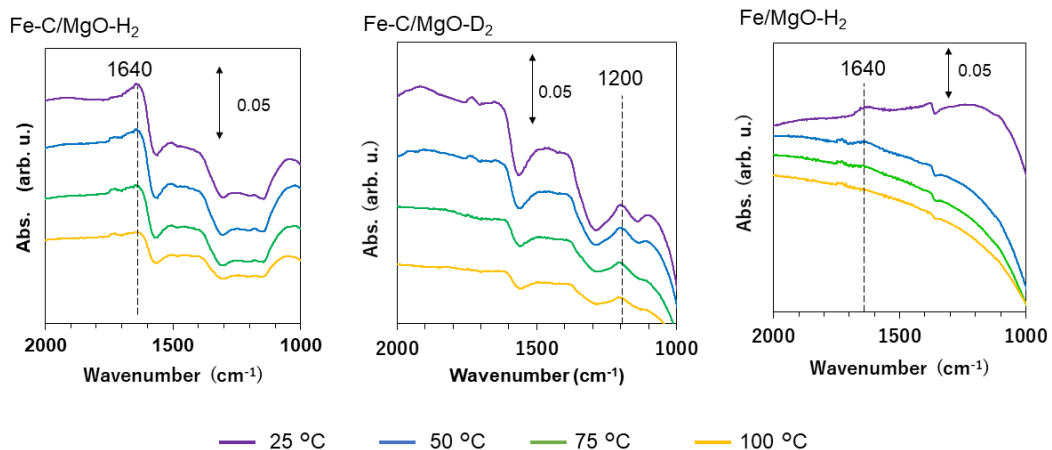


Fig. S10 H₂-pulse for Fe-C/MgO and Fe/MgO

Difference IR spectra from spectrum at 150 °C



Raw IR spectra

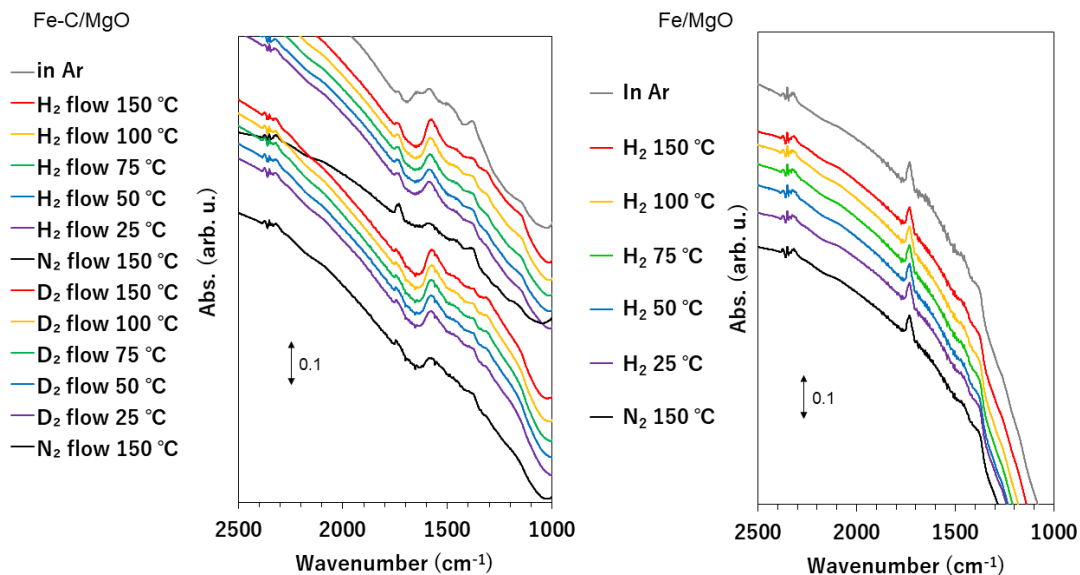


Fig. S11 H₂ and D₂-probed FT-IR spectra for Fe-C/MgO and Fe/MgO

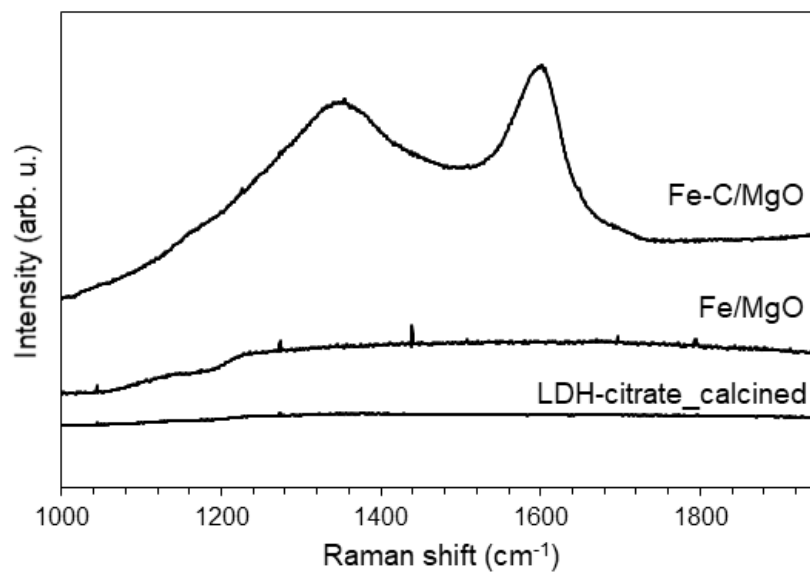


Fig. S12 Raman spectra for Fe-C/MgO, Fe/MgO, and LDH-citrate calcined in static air.

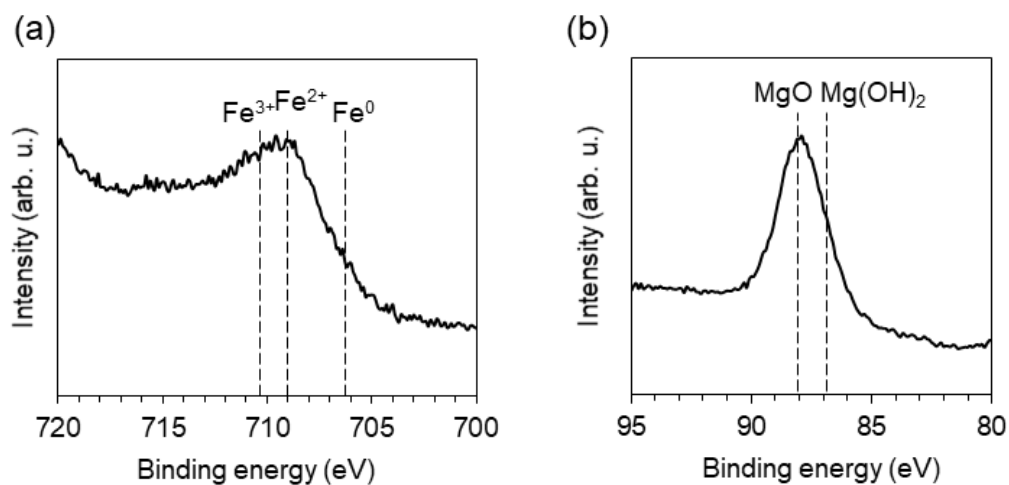


Fig. S13 (a) Fe 2p and (b) Mg 2s XPS spectra for Fe-C/MgO prepared by the reduction at 550 °C without exposure to air.

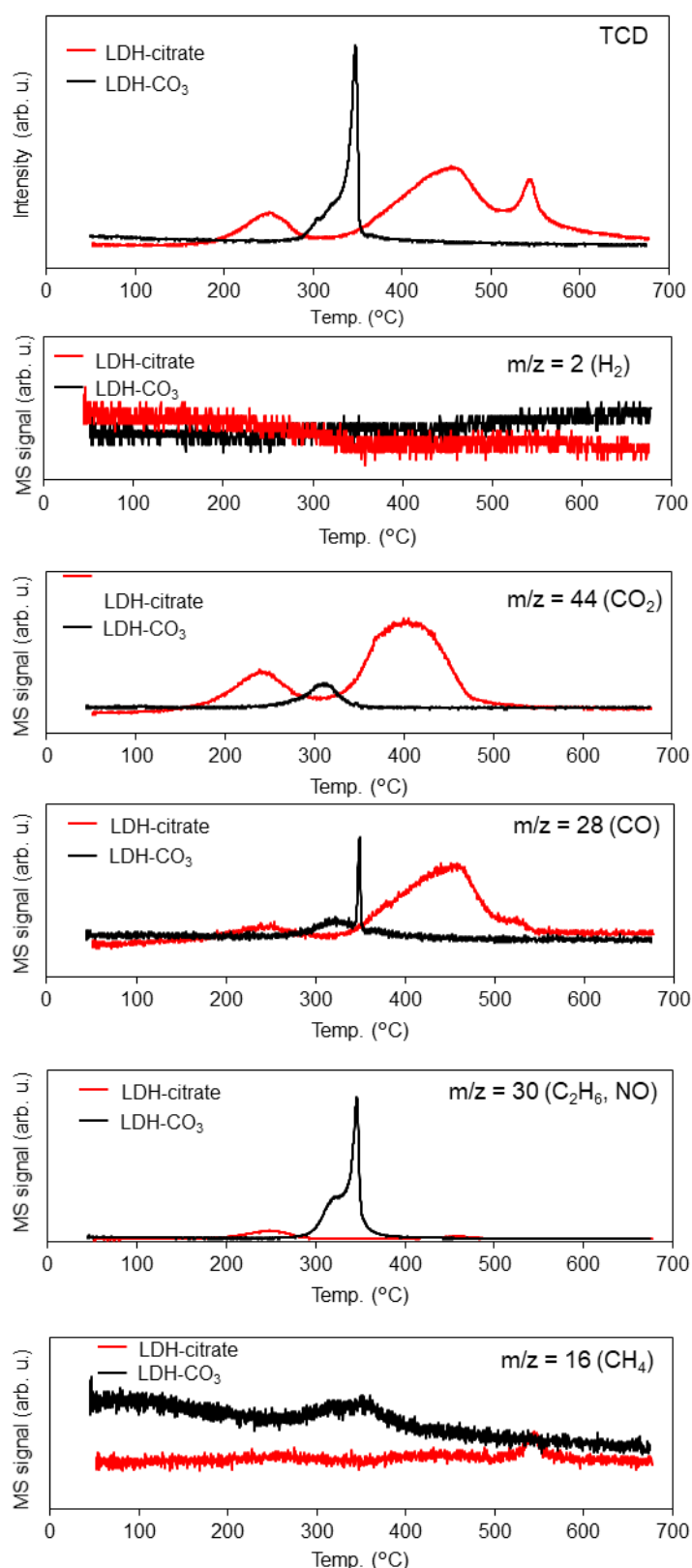


Fig. S14 TPR profiles for LDH-citrate and LDH-CO₃.

Conditions: sample 10 mg with H₂ flow (30 mL/min) from 50 to 700 °C

Hydrogen consumption cannot be observed because a large amount of gases is produced. Because the sample tube would break due to the generation of a large amount of gases, a small amount of sample was used for the measurement.

9. References

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