

Supplementary Materials

Magnetic porous chitosan-based palladium catalyst: A green, highly efficient and reusable catalyst for Mizoroki-Heck reaction in aqueous media

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1. Experimental Procedure

1.1. General

As received chitosan (CS) powder with average MW = 100000-300000 and the deacetylation degree of 70-85% (from Acros company) was used without further purification. All chemicals were commercial reagent grades and purchased from Merck and Aldrich. Polyethylene glycol (PEG, MW = 10000) was purchased from Aldrich. Thermogravimetric-diffraction thermal analysis (TG-DTA) was carried out using a thermal gravimetric analysis instrument (NETZSCH TG 209 F1 Iris) with a heating rate of 10 °C min⁻¹. XRD patterns were recorded by an EQUINOX 3000, X-ray diffractometer using Cu K α radiation. XPS (X-Ray photoelectron spectroscopy) data was recorded with 8025-BesTec twin anode XR3E2 X-ray source system. ¹H NMR (300 MHz) and ¹³C NMR (75 MHz) spectra were recorded using a Bruker AQS-300 Avance spectrometer. Transmission electron microscope, TEM (Zeiss - EM10C - 80 KV) was used to obtain TEM images. The scanning electron microscopy (SEM) images were obtained using a scanning electron microscope MIRA3\TESCAN-LMU. The magnetic measurement of samples were carried out in a vibrating sample magnetometer (VSM) (4 inch, Daghigh Meghnatis Kashan Co., Kashan, Iran) at room temperature. FT-IR spectra were obtained using a Shimadzu model FT-IR 8400 instrument. The Pd content of the complex was determined using inductively coupled plasma (ICP, Varian vista-mpx), and surface morphology of the catalyst was analyzed using Energy-dispersive-X-ray (MIRA3, TESCAN-LMU) equipped with EDX facility. Micro analytical data was collected by a Perkin-Elmer, USA, 2400C elemental analyzer. The N₂-sorption was carried out in a Belsorp-mini-BEL Japan, Inc. at 298 K.

1.2. Preparation of porous chitosan-thienyl imine support, PCS-TI (1) and its characterization

In a 200 mL round-bottom flask equipped with a magnetic stirring bar, chitosan (1 g) was allowed to dissolve slowly in aqueous acetic acid solution (0.87 M, 100 mL), for 1 h at room temperature; then PEG (1 g) was added to the above flask and stirred for 10 min, followed by addition of 2-thiophenecarbaldehyde (2.24 g, 20 mmol) and the mixture was stirred for 24 h under N₂ atmosphere. The reaction mixture was dripped into a 1 N NaOH aqueous solution (250 mL) through a needle of 0.9 mm diameter. The gelatine like CS-TI was collected and washed with hot distilled water until neutral (pH ~ 7), followed by extraction of the PEG component with about 2 liters of hot water (Scheme 1). Similarly, the bead chitosan-supported ligand (BCS-TI) was prepared by the same protocol in the absence of PEG. Also, the normal chitosan-supported ligand (NCS-TI) was produced by mixing chitosan and 2-thiophenecarbaldehyde in EtOH (10 mL).

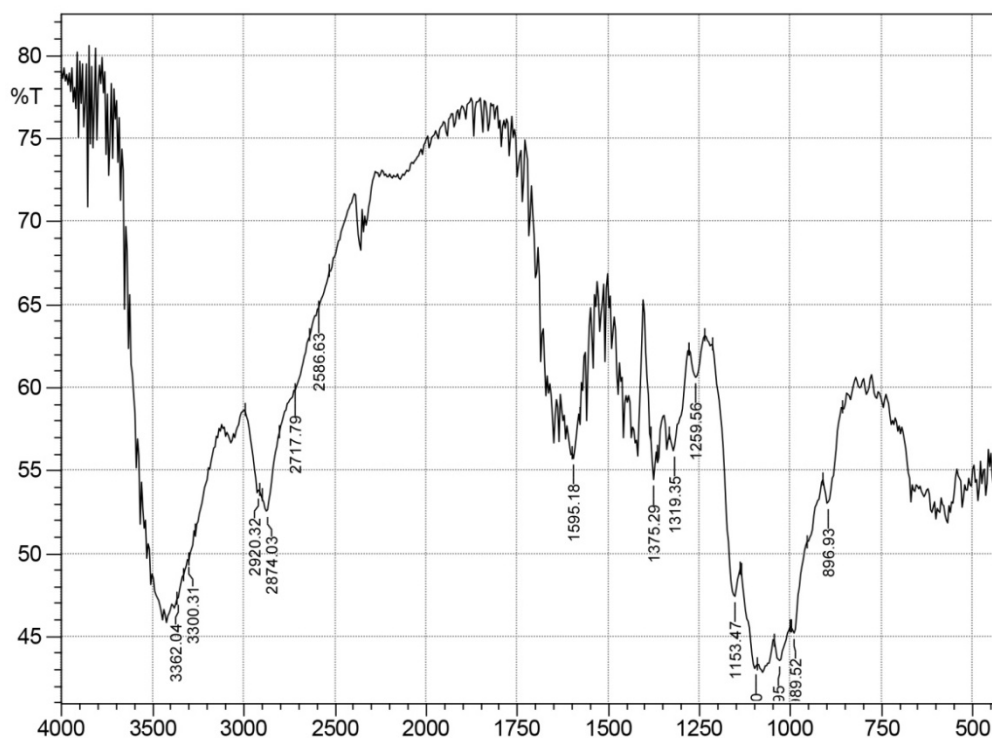


Figure S1. IR (KBr disc) spectrum of chitosan.

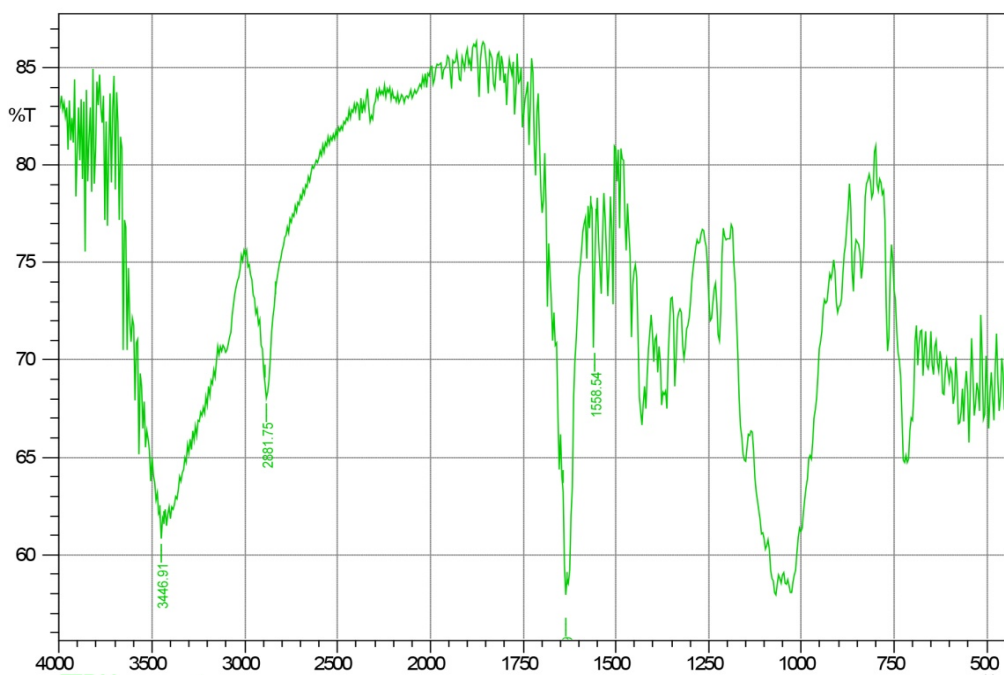


Figure S2. IR (KBr disc) spectrum of PCS-TI (1).

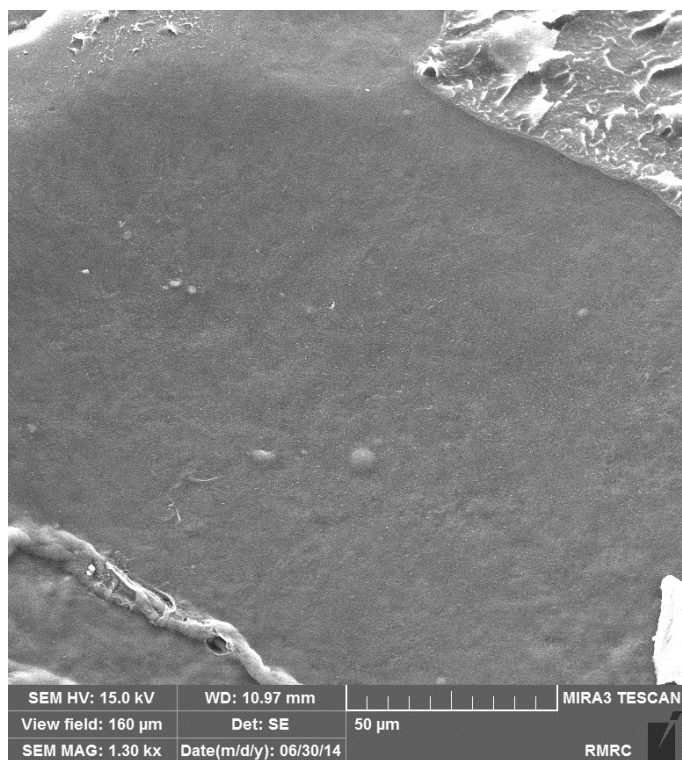


Figure S3. SEM image of NCS-TI.

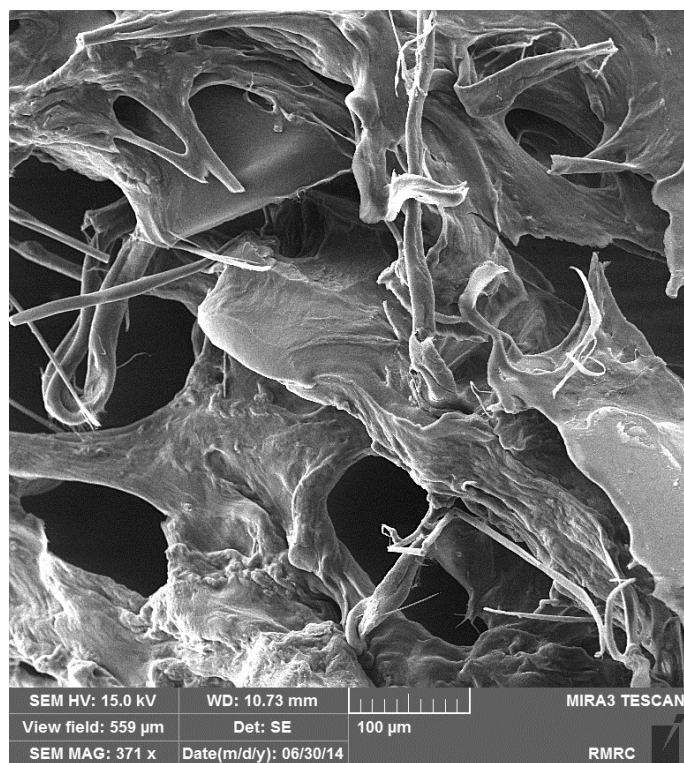
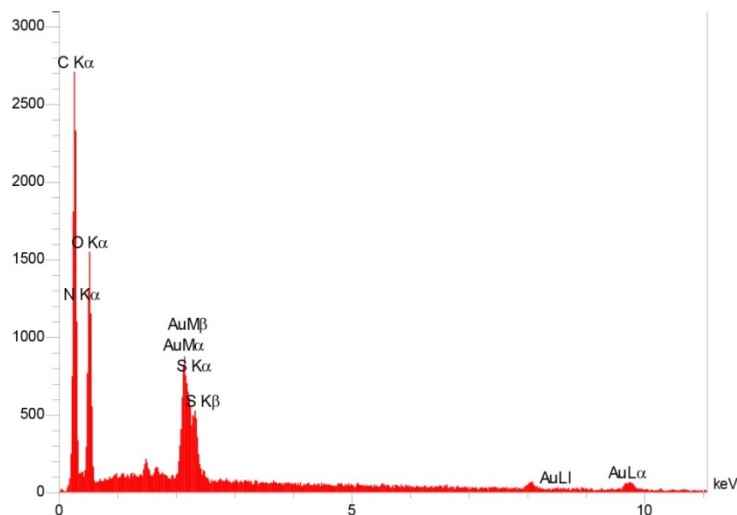


Figure S4. SEM image of PCS-TI (1).

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Quantitative Results

Elt	LiLine	Int	Error	K	Kr	W%	A%	ZAF	Formula	Ox%	Pk/Bg	Class	LConf	HConf	Cat#
C	Ka	194.8	21.9233	0.4983	0.2071	41.35	59.01	0.4891		0.00	78.09	A	41.45	43.25	0.00
N	Ka	8.6	21.9233	0.0296	0.0123	7.60	9.18	0.1620		0.00	5.20	A	6.83	8.37	0.00
O	Ka	121.3	21.9233	0.1546	0.0642	26.06	28.02	0.2374		0.00	37.02	A	26.33	27.79	0.00
S	Ka	39.4	1.4186	0.0236	0.0098	3.17	1.92	0.8365		0.00	4.33	A	1.12	1.23	0.00
Au	La	5.9	0.4941	0.2939	0.1222	21.81	1.87	0.5599		0.00	3.48	B	19.15	24.48	0.00
				1.0000	0.4156	100.00	100.00			0.00					0.00

Figure S5. EDX spectrum of PCS-TI (1).

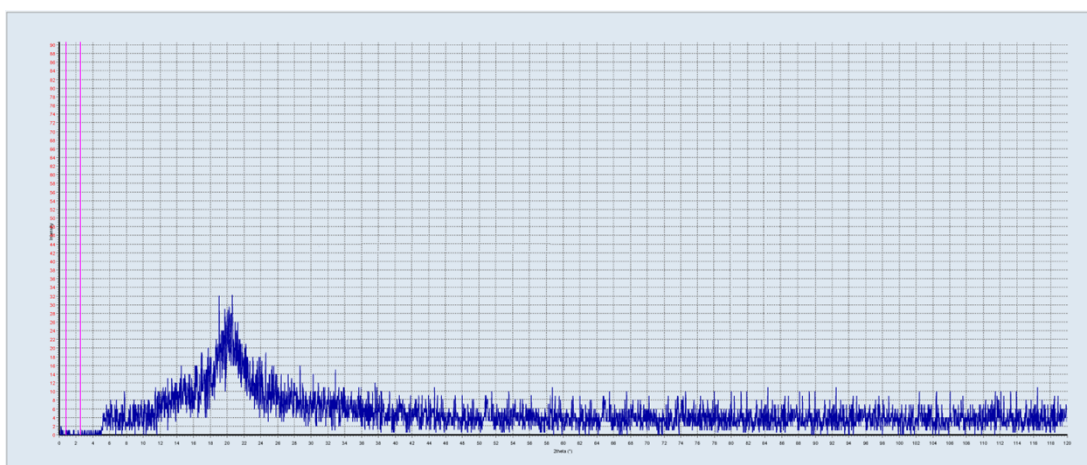


Figure S6. Powder XRD pattern of PCS-TI (1).

1.3. Preparation and characterization of porous chitosan-thienyl imine Pd complex,

PCS-TI/Pd (2)

In a 50 mL round-bottom flask, PdCl₂ (0.088 g, 0.5 mmol) was added to PCS-TI (1) in EtOH (30 mL). The mixture was heated at reflux for 24 h. The product formed was filtered off, washed with EtOH and finally dried in vacuum for 24 h at 50 °C (Scheme 1). Similarly, the bead and normal chitosan-thienyl Pd complexes (BCS-TI/Pd and NCS-TI/Pd) were prepared by the same protocol.

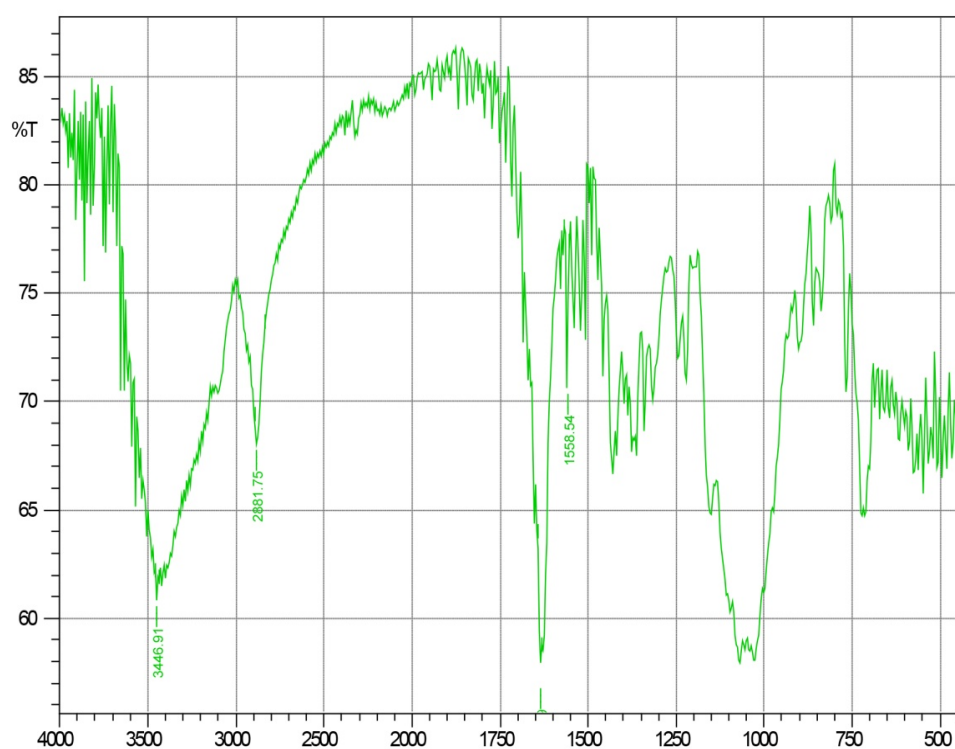


Figure S7. IR (KBr) spectrum of PCS-TI/Pd (2).

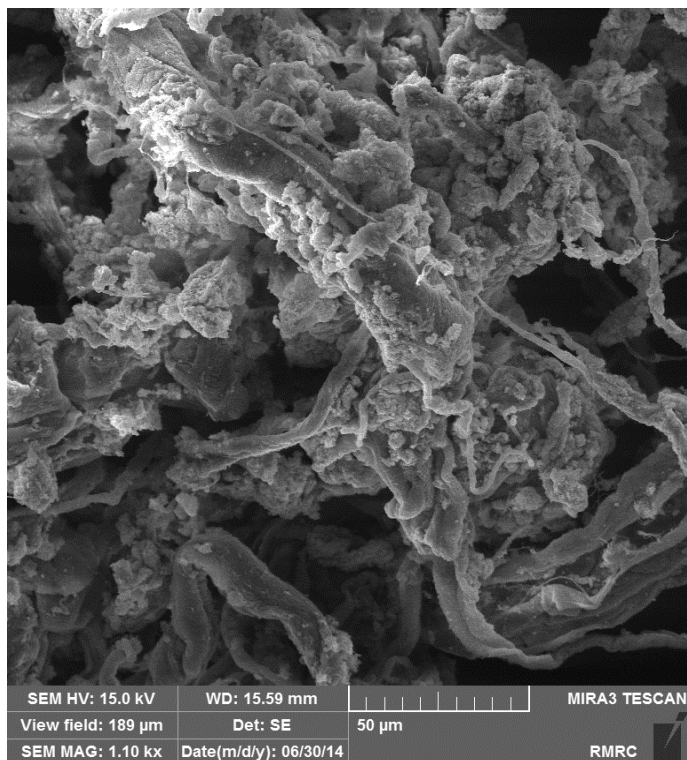


Figure S8. SEM image of PCS-TI/Pd (2).

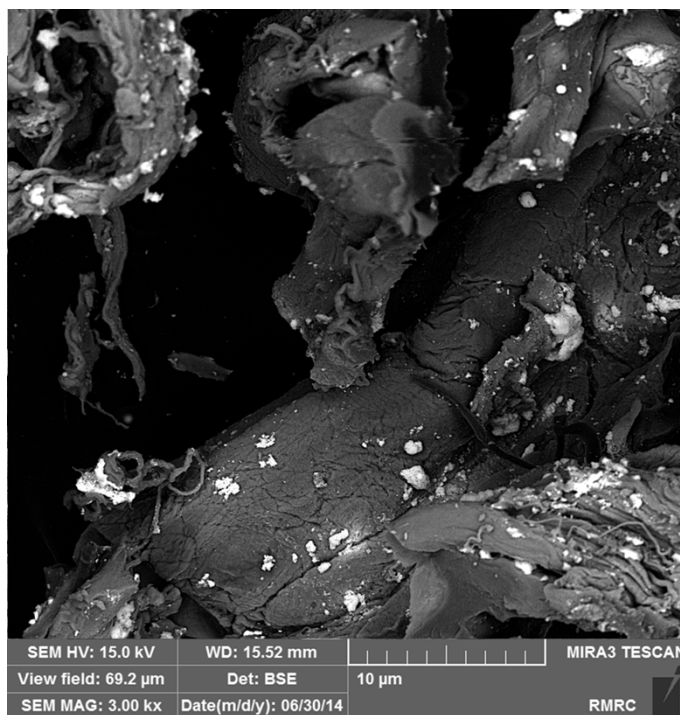
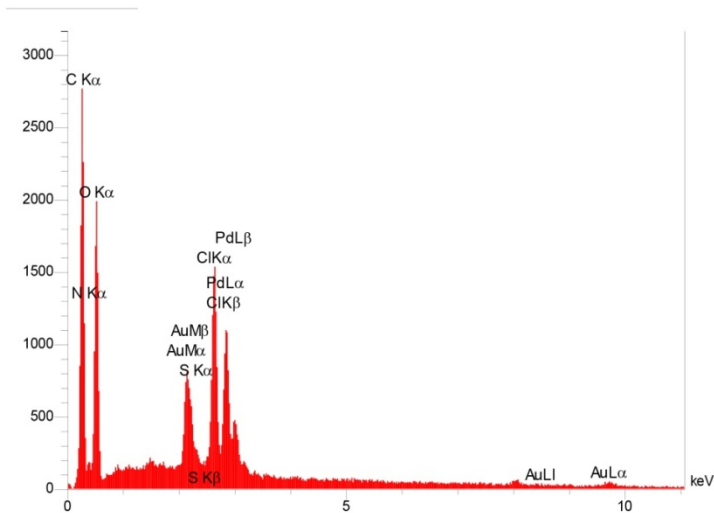


Figure S9. SEM image of PCS-TI/Pd (2) (distribution of palladium).

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Quantitative Results

Elt	Line	Int	Error	K	Kr	W%	A%	ZAF	Formula	Ox%	Pk/Bg	Class	LConf	HConf	Cat#
C	Ka	197.6	29.0647	0.4367	0.1679	39.37	54.29	0.4212		0.00	119.85	A	39.03	40.72	0.00
N	Ka	13.2	29.0647	0.0393	0.0151	8.92	10.58	0.1523		0.00	7.25	A	9.11	10.74	0.00
O	Ka	146.3	29.0647	0.1611	0.0620	29.09	30.15	0.2100		0.00	53.19	A	28.77	30.22	0.00
S	Ka	11.9	0.8547	0.0062	0.0024	2.27	1.14	0.8706		0.00	3.94	B	0.25	0.30	0.00
Cl	Ka	163.2	0.8547	0.0935	0.0359	4.28	1.98	0.8391		0.00	13.11	A	4.18	4.38	0.00
Pd	La	107.7	0.8547	0.1397	0.0537	7.35	1.13	0.7307		0.00	11.44	A	7.14	7.57	0.00
Au	La	2.9	0.3529	0.1236	0.0475	8.80	0.73	0.5403		0.00	2.63	B	7.26	10.34	0.00
				1.0000	0.3846	100.00	100.00			0.00					0.00

Figure S10. EDX spectrum of PCS-TI/Pd (2).

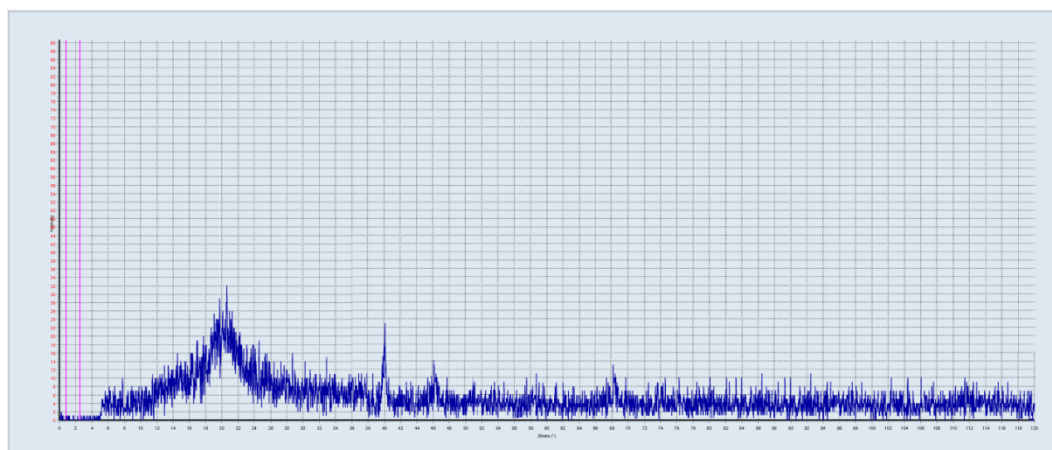


Figure S11. Powder XRD pattern of PCS-TI/Pd (2).

1.4. Preparation and characterization of cross-linked magnetic porous chitosan-thienyl imine Pd complex MPCS-TI/Pd (3)

PCS-TI/Pd (2) was added to aqueous acetic acid solution (0.17 M, 30 mL); a suspension of Fe_3O_4 (0.5 g) in glyoxal (3 mL) and EtOH (5 mL) was then added dropwise to the above mixture. More glyoxal (1.5 mL) was added and the reaction mixture was mechanically stirred for 1 h at room temperature. The product formed was collected, washed with water, ethanol, and then dried in vacuum at 50 °C for 24 h (Scheme 1). Similarly, magnetic bead and normal catalysts (MBCS-TI/Pd, MNCS-TI/Pd) were prepared by the same protocol.

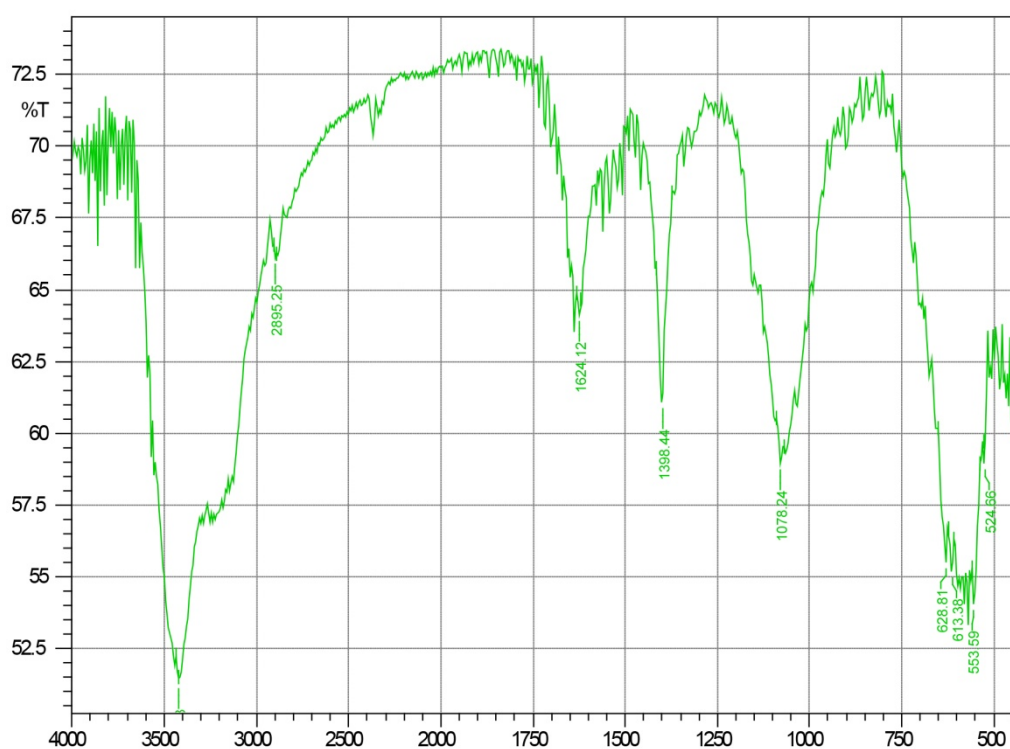


Figure S12. IR (KBr) spectrum of MPCS-TI/Pd (3).

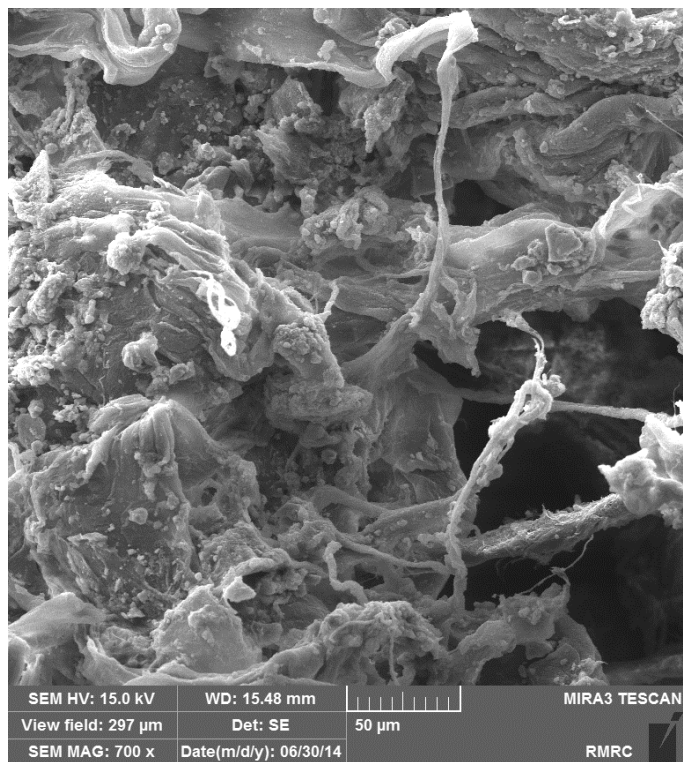


Figure S13. SEM image of MPCS-TI/Pd (3).

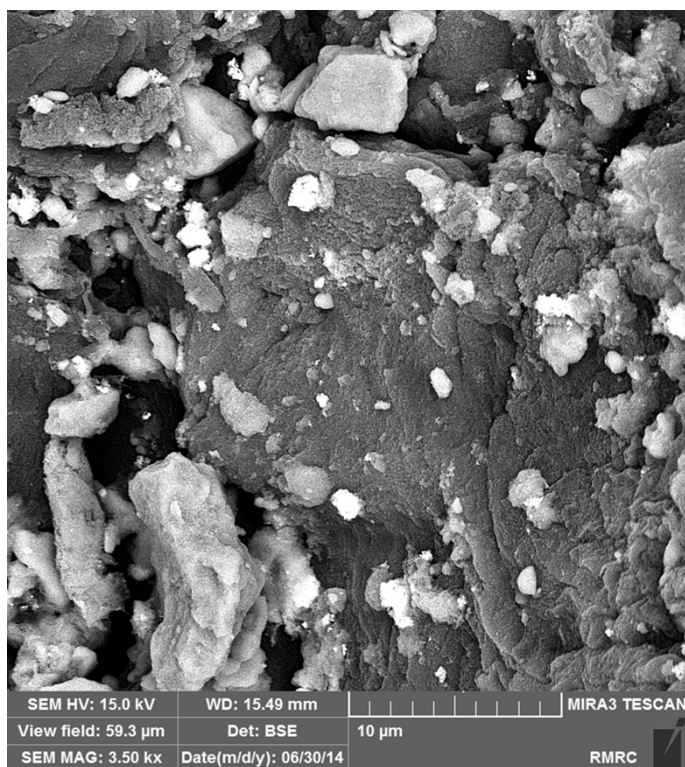


Figure S14. SEM image of MPCS-TI/Pd (3) (distribution of nanoparticles).

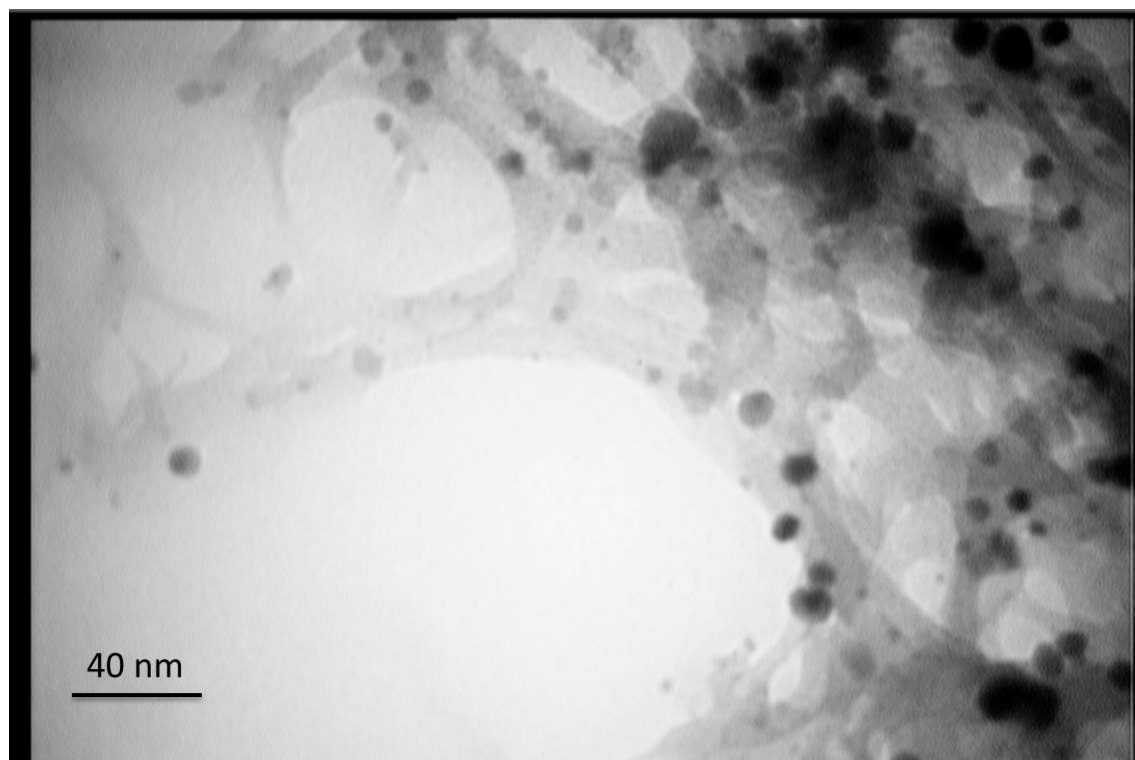
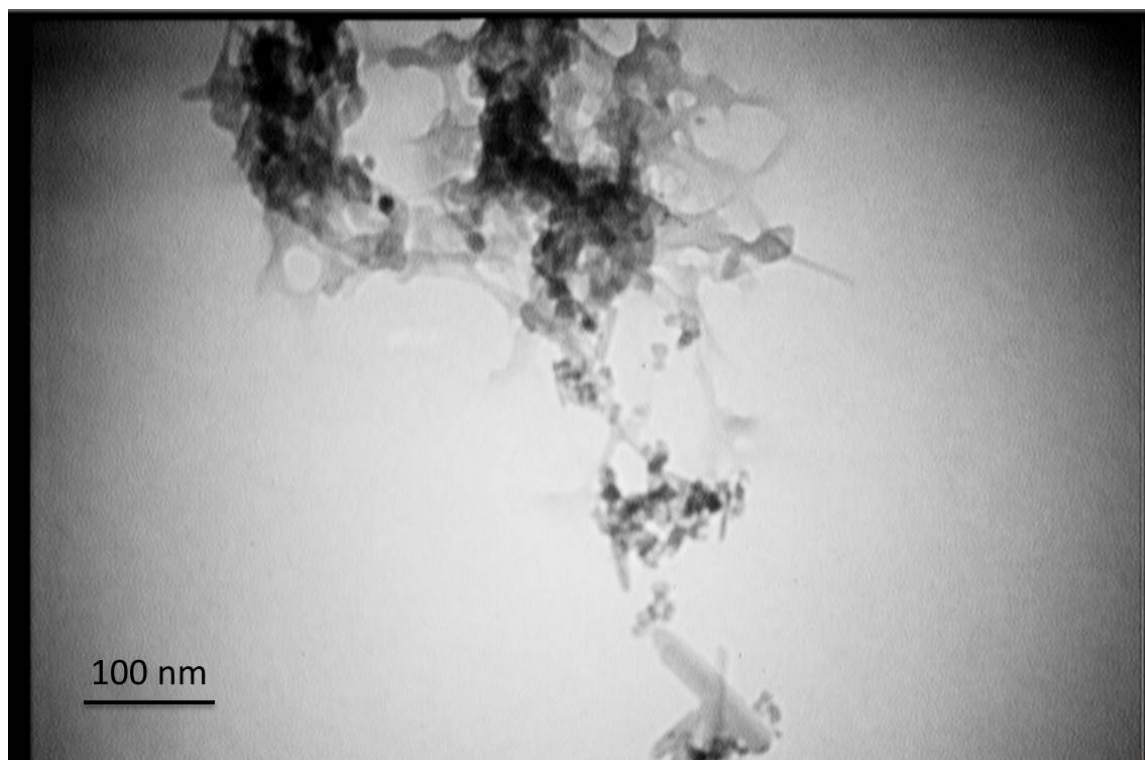


Figure S15. TEM image of MPCS-TI/Pd (**3**) before reaction.

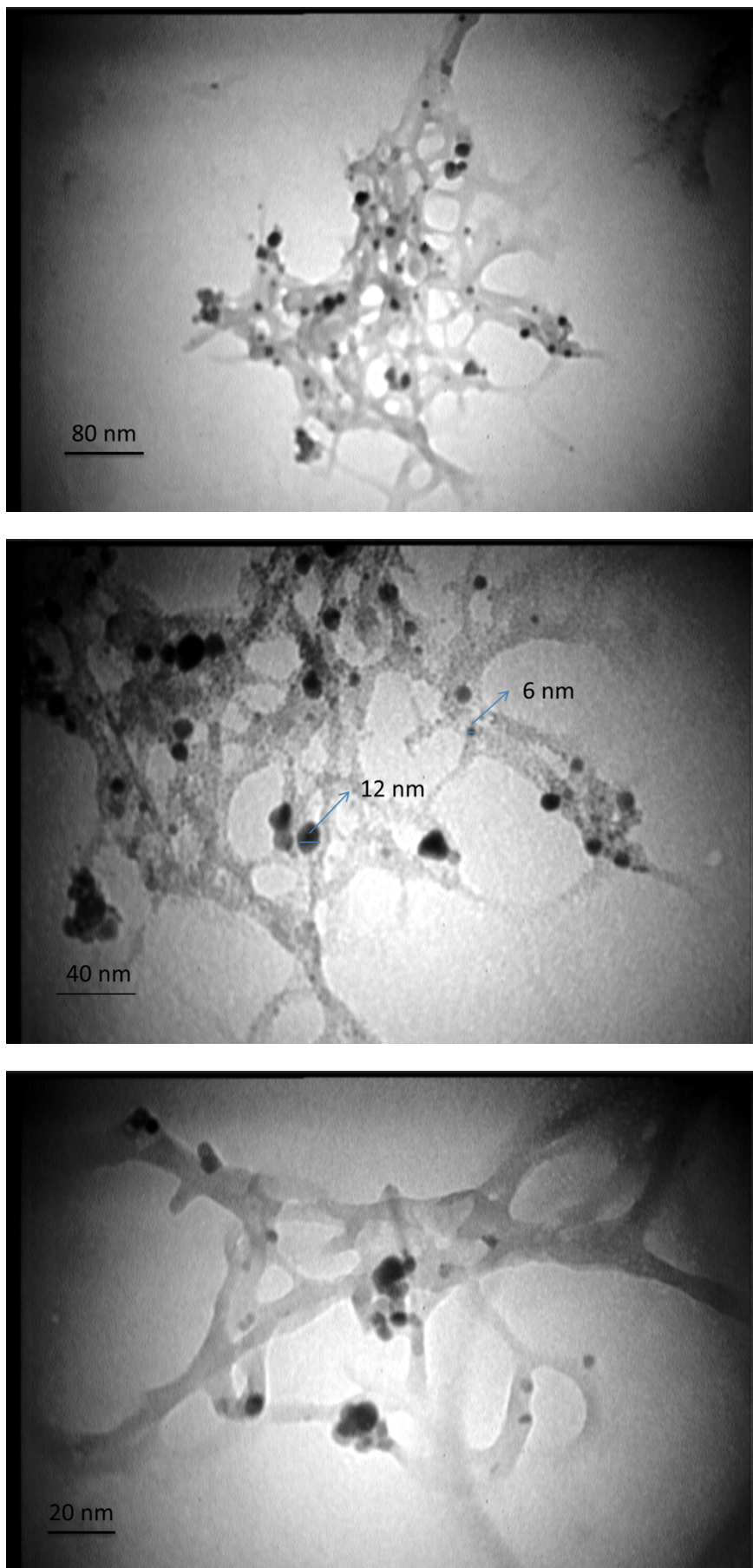
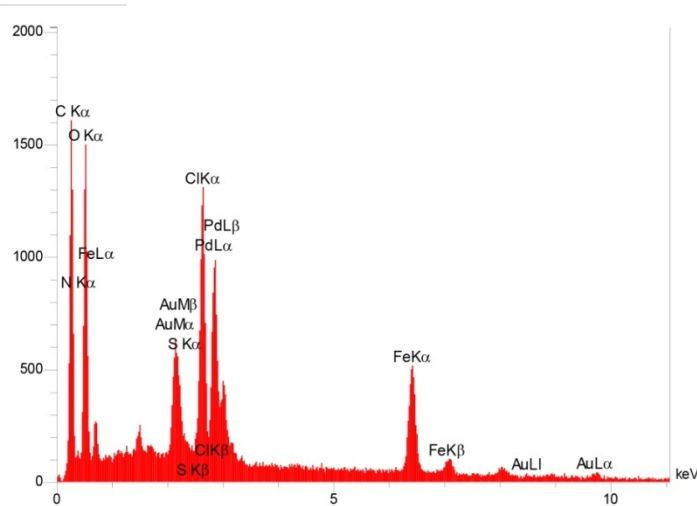


Figure S16. TEM image of MPCS-Ti/Pd (**3**) after eight reaction runs.

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Quantitative Results

Elt	Line	Int	Error	K	Kr	W%	A%	ZAF	Formula	Ox%	Pk/Bg	Class	LConf	HConf	Cat#
C	Ka	114.7	11.9980	0.3065	0.1312	34.50	51.48	0.3780		0.00	75.47	A	33.73	35.66	0.00
N	Ka	8.6	11.9980	0.0309	0.0132	6.99	9.16	0.1655		0.00	5.41	A	7.17	8.80	0.00
O	Ka	111.8	11.9980	0.1488	0.0637	27.73	30.90	0.2296		0.00	37.07	A	26.95	28.52	0.00
S	Ka	6.2	1.2195	0.0039	0.0017	2.19	1.10	0.8761		0.00	3.43	B	0.17	0.21	0.00
Cl	Ka	136.2	1.2195	0.0943	0.0404	4.73	2.38	0.8534		0.00	11.60	A	4.61	4.85	0.00
Fe	Ka	75.1	0.5351	0.1738	0.0744	9.02	2.88	0.8252		0.00	10.61	A	8.71	9.33	0.00
Pd	La	98.4	1.2195	0.1543	0.0660	8.87	1.49	0.7440		0.00	10.81	A	8.61	9.14	0.00
Au	La	1.7	0.2885	0.0876	0.0375	6.78	0.61	0.5533		0.00	2.29	B	5.22	8.33	0.00
				1.0000	0.4280	100.00	100.00			0.00					0.00

Figure S17. EDX spectrum of MPCs-Ti/Pd (3).

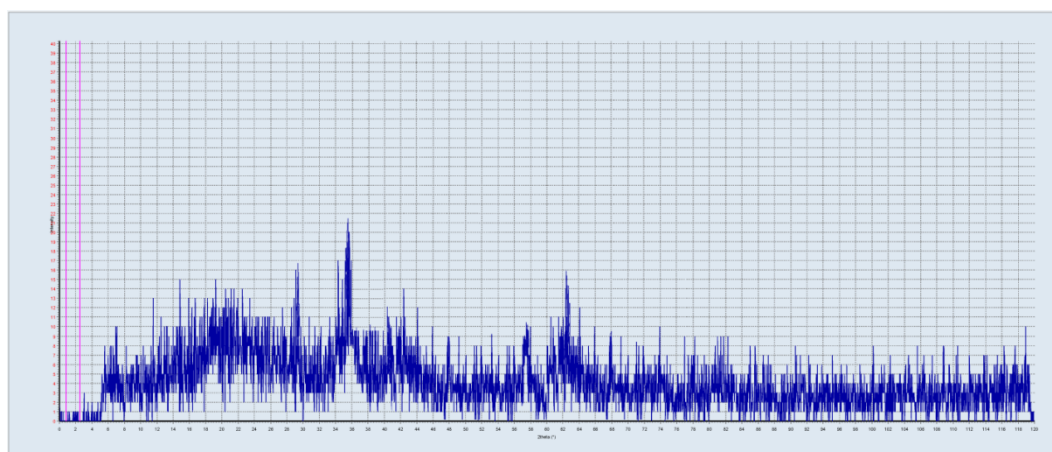


Figure S18. Powder XRD pattern of MPCs-Ti/Pd (3).

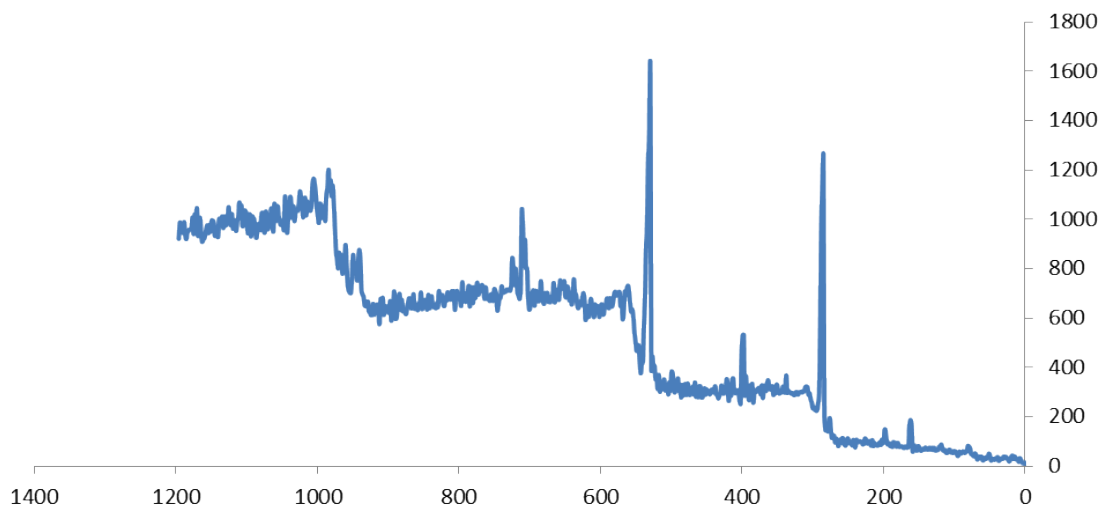


Figure S19. XPS spectrum of the MPCS-TI/Pd (**3**), before reaction.

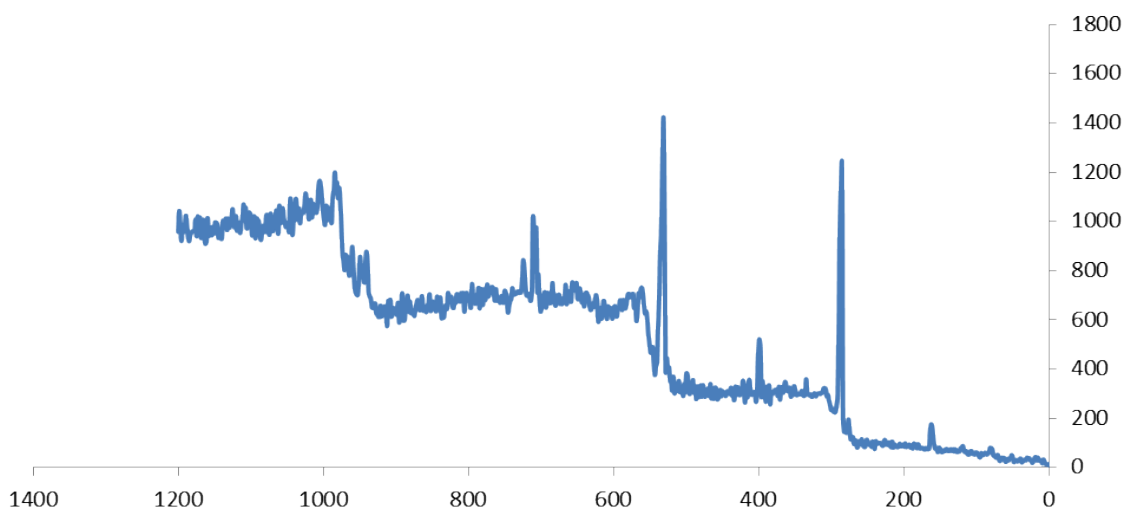


Figure S20. XPS spectrum of the MPCS-TI/Pd (**3**), after reaction.

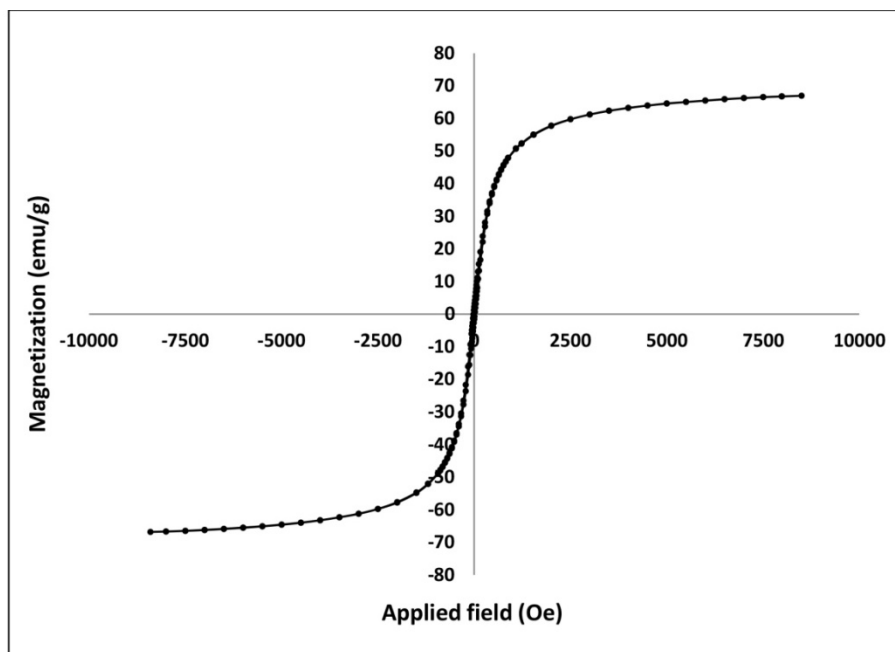


Figure S21. VSM curve for the magnetic Fe₃O₄ nanoparticles.

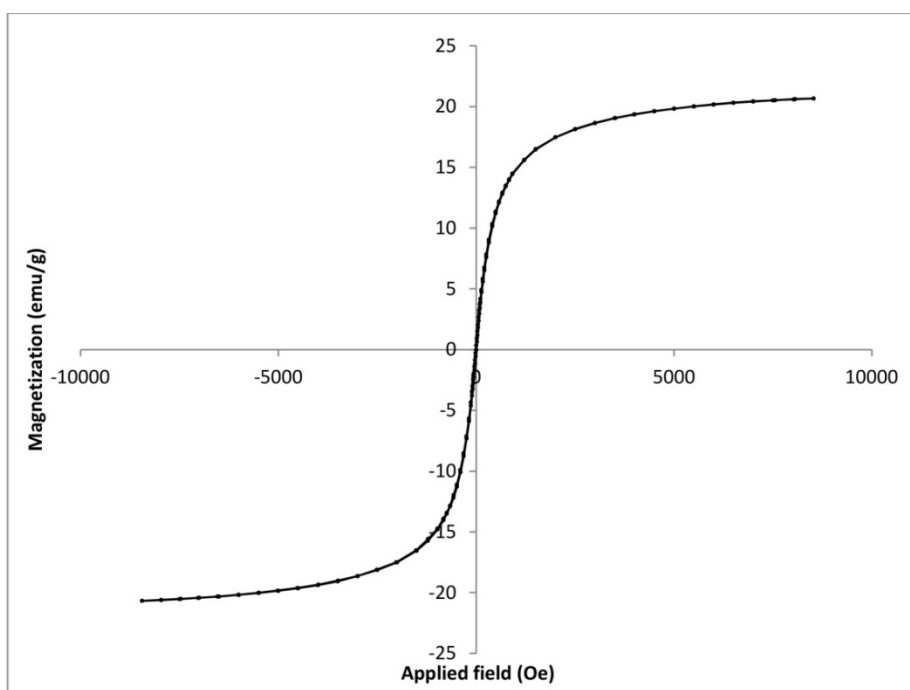


Figure S22. VSM curve for the magnetic MPCS-TI/Pd (3).

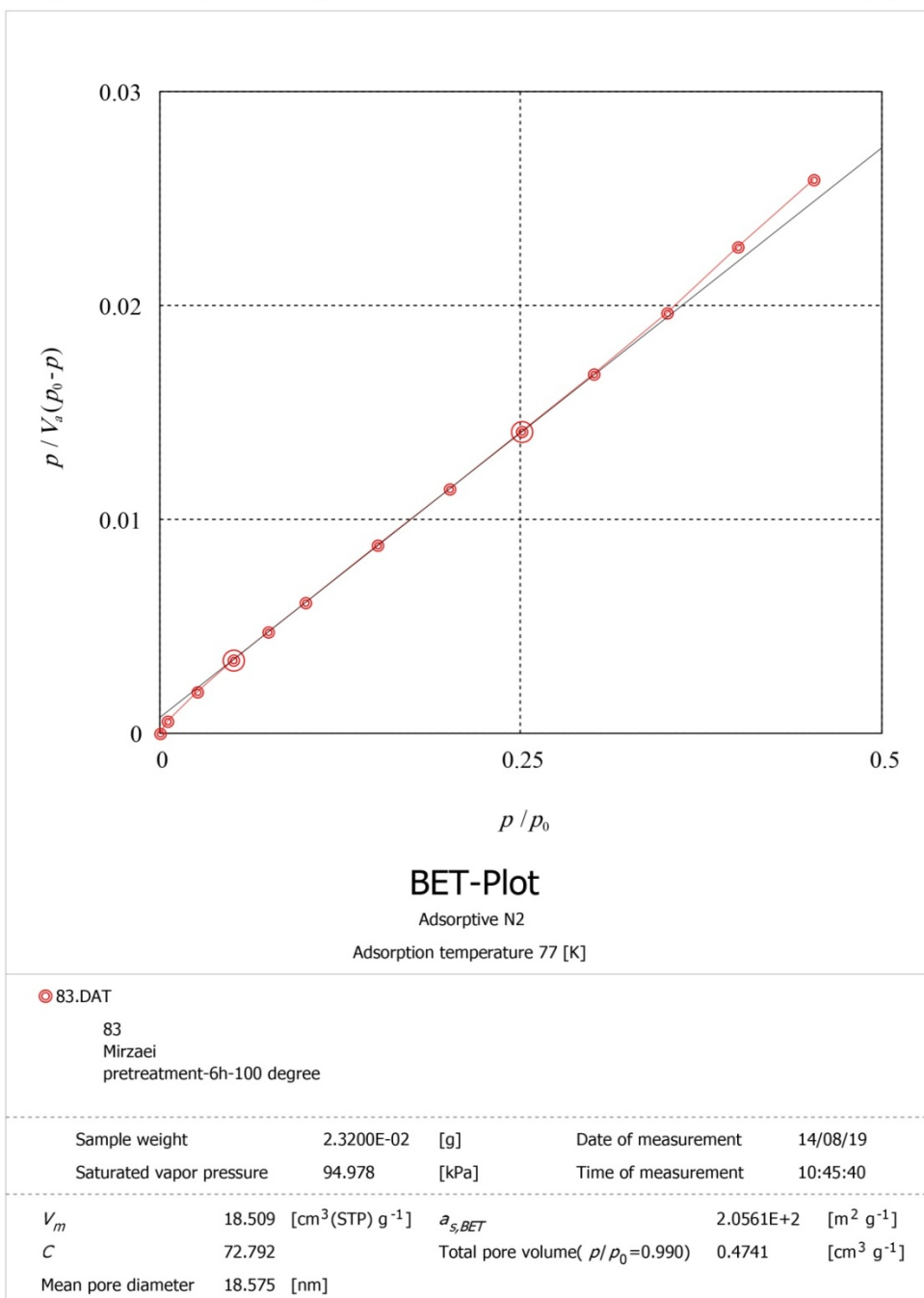


Figure S23. BET diagram of MPCs-TI/Pd (3).

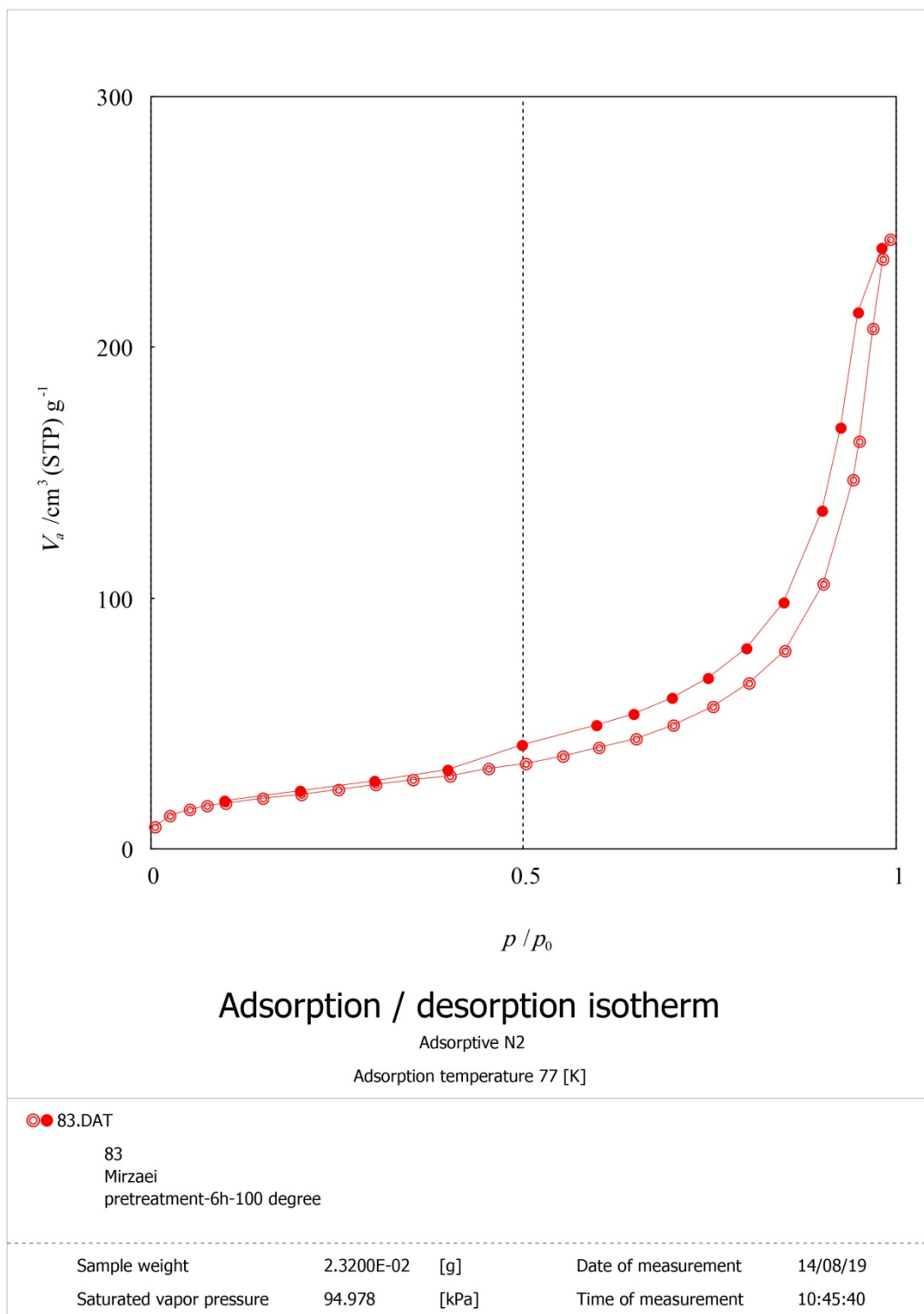


Figure S24. N₂ adsorption-desorption isotherm of MPCs-TI/Pd (3).

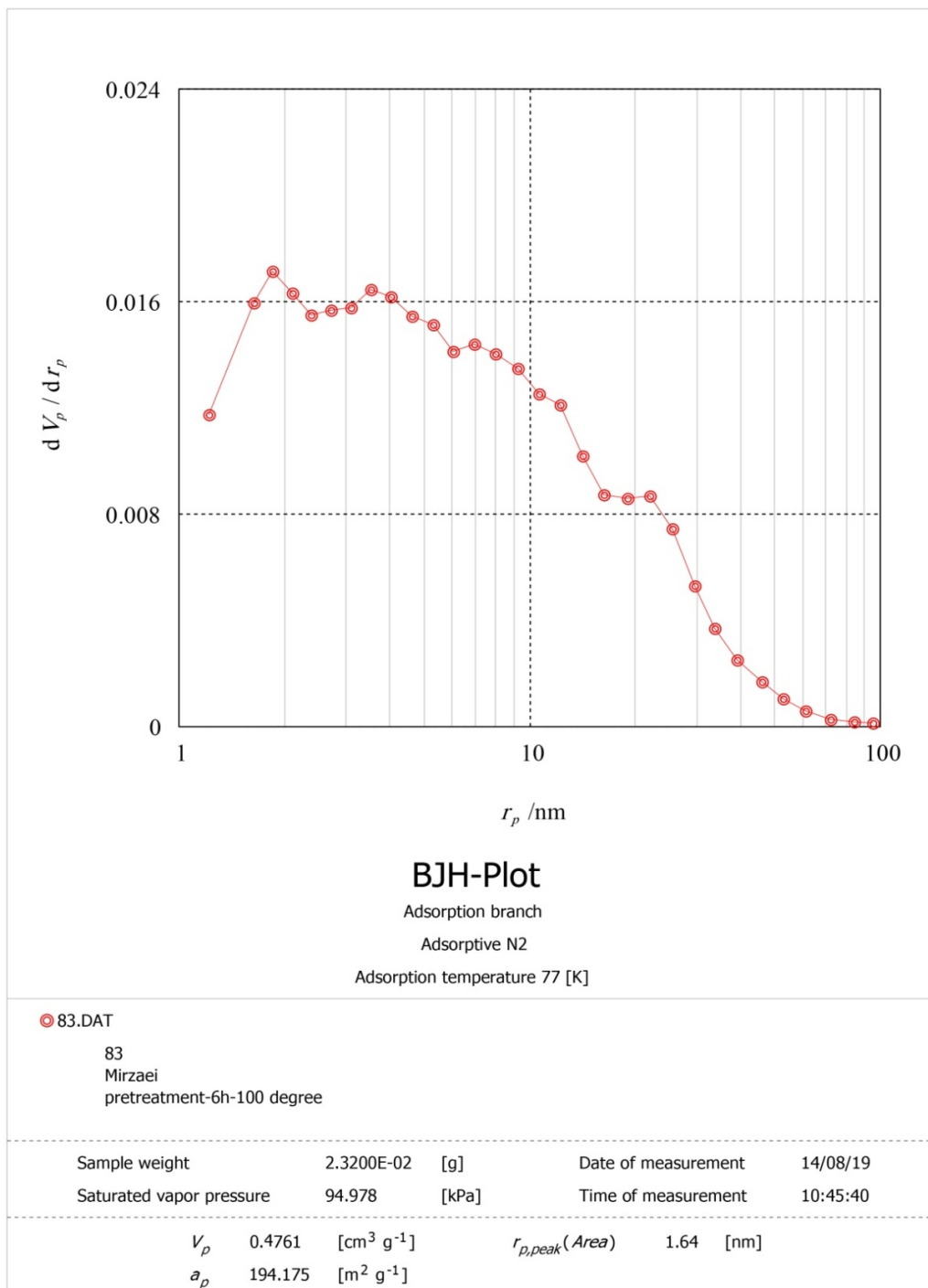


Figure S25. BJH diagram of MPCs-TI/Pd (3).

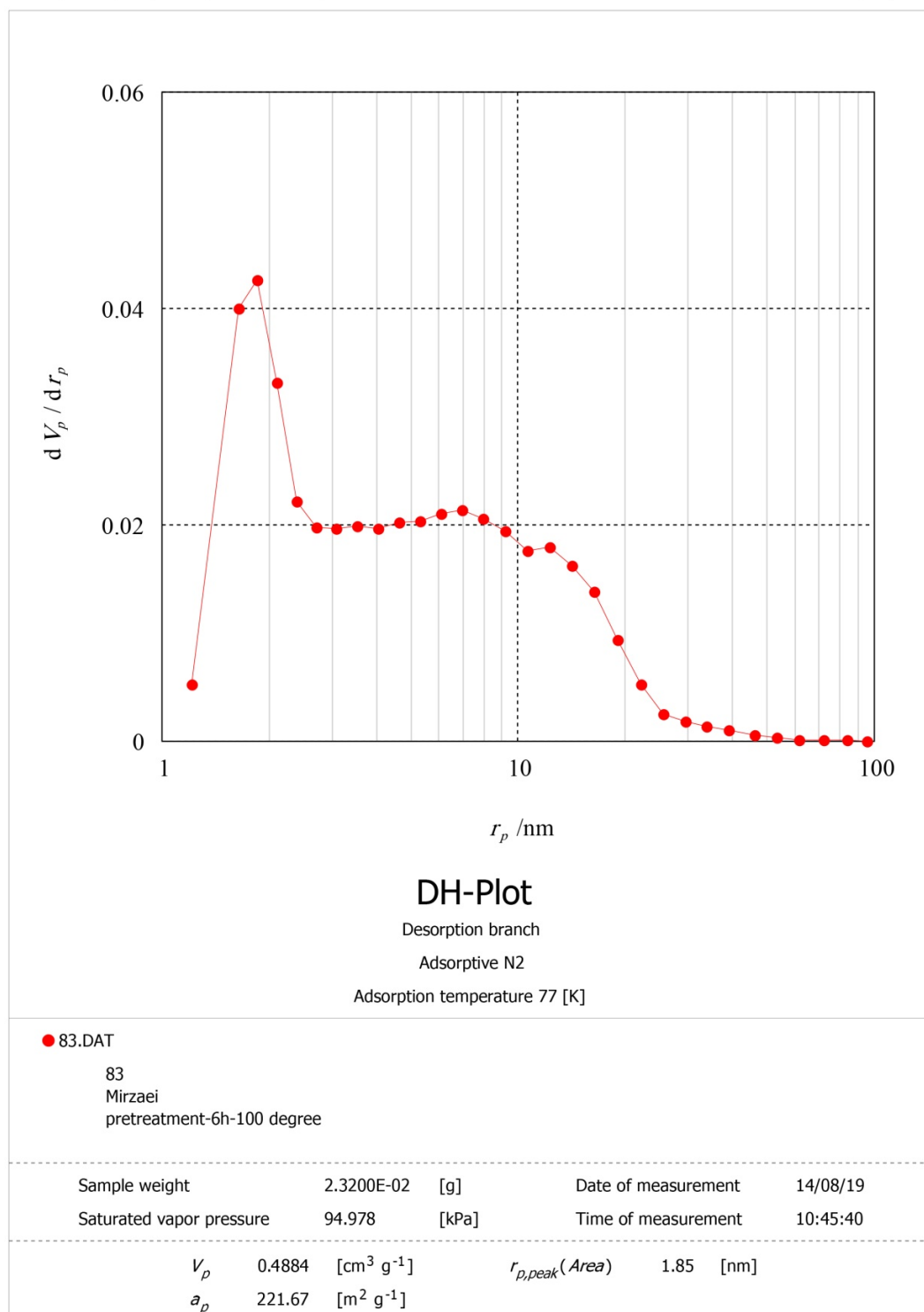


Figure S26. DH diagram of MPCS-TI/Pd (3).

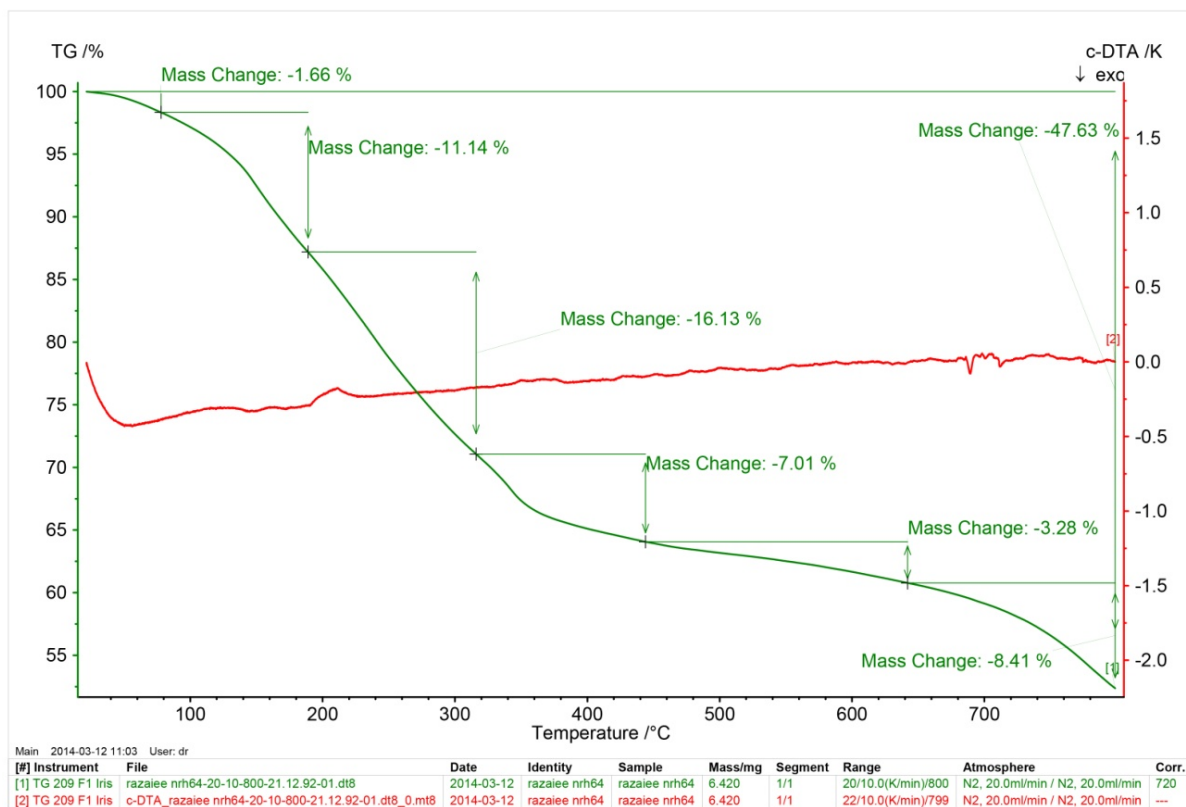
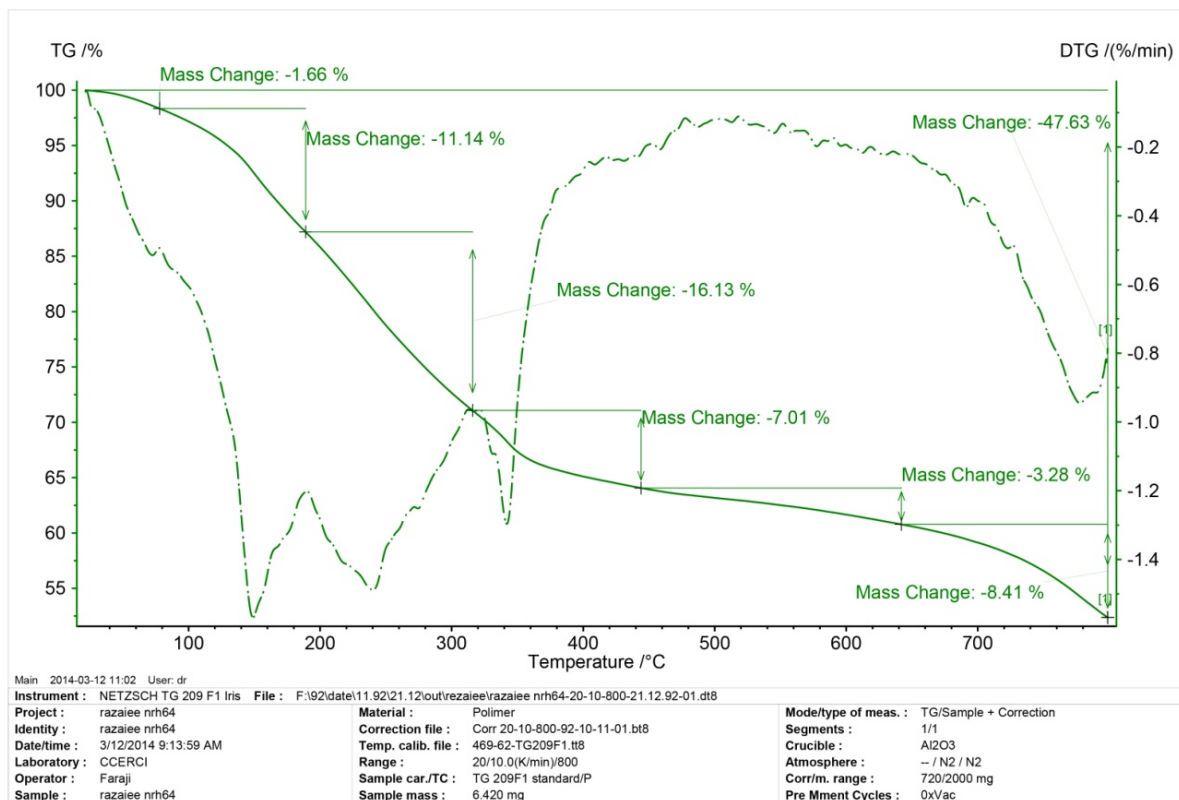


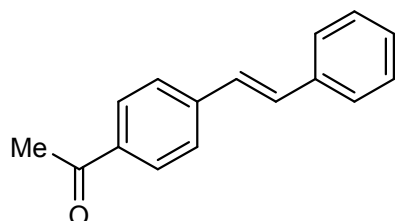
Figure S27. TG and DTA curves of MPCS-TI/Pd (3).

1.5. General procedure for Mizoroki-Heck coupling reaction

A mixture of aryl halide (1.0 mmol), alkene (1.3 mmol), triethylamine (2 mmol), TBAB (0.5 mmol) H₂O/DMF (v/v = 2:1, 3 mL), and the catalyst (0.001 mmol, 0.1 mol% Pd) was stirred at 110 °C for an appropriate time under aerial condition. The progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was cooled to room temperature, poured into H₂O (10 mL), and the catalyst was separated by magnetic decantation. In the case of acrylic acid, aqueous solution of Na₂CO₃ (10 mL, 3% w/v) was added; after separation of the catalyst, 1 N aq. HCl (5 mL) was added, and the product was extracted with ethyl acetate (3 × 10 mL). The combined organic extracts were washed with brine (2 × 10 mL), dried (MgSO₄), and concentrated in vacuum. The crude product was further purified by preparative TLC (silica gel) using *n*-hexane–EtOAc (9:1) to afford the desired product.

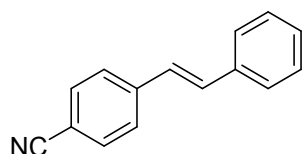
2. Spectral data for Table 2

(*E*)-4-Acetylstilbene (Table 2, 6b)¹



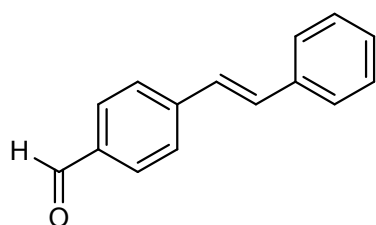
Pale-yellow solid; mp 141–143 °C; IR (KBr): $\nu = 1678.4 \text{ cm}^{-1}$; ¹H NMR (300 MHz, CDCl₃): $\delta = 7.97$ (d, $J = 8.3 \text{ Hz}$, 2H), 7.60 (d, $J = 8.4 \text{ Hz}$, 2H), 7.55 (d, $J = 7.5 \text{ Hz}$, 2H), 7.39 (t, $J = 7.1 \text{ Hz}$, 2H), 7.31–7.33 (m, 1H), 7.24 (d, $J = 14.3 \text{ Hz}$, 1H), 7.14 (d, $J = 16.3 \text{ Hz}$, 1H), 2.62 (s, 3H). ¹³C NMR (75 MHz, CDCl₃): $\delta = 197.5, 142.0, 136.7, 135.9, 131.4, 128.9, 128.8, 128.3, 127.4, 126.8, 126.5, 26.6$.

(*E*)-4-Cyanostilbene (Table 2, 6c)¹



White solid; mp 116–118 °C; IR (KBr): $\nu = 2231.9 \text{ cm}^{-1}$; ¹H NMR (300 MHz, CDCl₃): $\delta = 7.53$ – 7.66 (m, 6H), 7.27– 7.43 (m, 3H), 7.23 (d, $J = 16.4 \text{ Hz}$, 1H), 7.09 (d, $J = 16.3 \text{ Hz}$, 1H); ¹³C NMR (75 MHz, CDCl₃): $\delta = 141.8, 136.3, 132.5, 132.4, 128.9, 128.7, 126.94, 126.9, 126.7, 119.1, 110.6$.

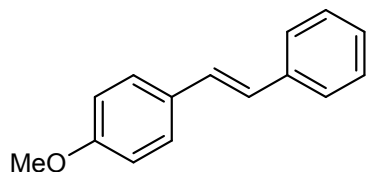
(*E*)-4-Formylstilbene (Table 2, 6d)²



Pale-yellow solid; mp 113–116 °C; IR (KBr): $\nu = 1700.2 \text{ cm}^{-1}$; ¹H NMR (300 MHz, CDCl₃): $\delta = 10.0$ (s, 1H), 7.88 (d, $J = 7.8 \text{ Hz}$, 2H), 7.66 (d, $J = 7.8 \text{ Hz}$, 2H), 7.56 (d, $J = 7.2 \text{ Hz}$, 2H), 7.31– 7.42 (m,

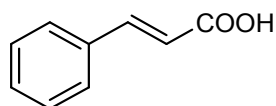
3H), 7.28 (d, $J = 16.3$ Hz, 1H), 7.15 (d, $J = 16.3$ Hz, 1H); ^{13}C NMR (75 MHz, CDCl_3): $\delta = 191.7$, 143.4, 136.5, 135.3, 132.2, 130.3, 128.9, 128.5, 127.3, 126.9.

(E)-4-Methoxystilbene (Table 2, 6f)³



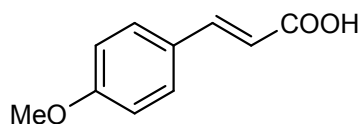
White solid; mp 134–136 °C; ^1H NMR (300 MHz, CDCl_3): $\delta = 7.45$ -7.52 (m, 4H), 7.36 (t, $J = 7.7$ Hz, 2H), 7.22-7.28 (m, 1H), 7.09 (d, $J = 16.3$ Hz, 1H), 6.99 (d, $J = 16.3$ Hz, 1H), 6.89-6.94 (m, 2H), 3.85 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3): $\delta = 159.3$, 137.6, 130.1, 128.8, 128.2, 127.7, 127.2, 126.6, 126.3, 114.1, 55.3.

Cinnamic acid (Table 2, 6k)⁴



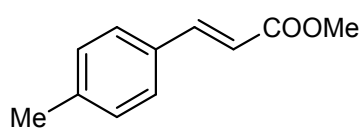
White solid; IR (KBr): $\nu = 2963.1$, 1697.9 cm^{-1} ; m.p. 131-133 °C; ^1H NMR (300 MHz, CDCl_3): $\delta = 11.53$ (s, 1H), 7.82 (d, $J = 16.0$ Hz, 1H), 7.54-7.59 (m, 2H), 7.41-7.45 (m, 3H), 6.48 (d, $J = 16.0$ Hz, 1H); ^{13}C NMR (75 MHz, CDCl_3): $\delta = 172.5$, 147.1, 134.0, 130.8, 128.97, 128.4, 117.3.

(E)-4-Methoxycinnamic acid (Table 2, 6l)⁴



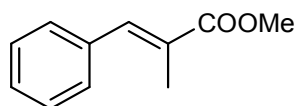
White solid; m.p. 171-174 °C; IR (KBr): $\nu = 2938.2$, 1686.5 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3): $\delta = 11.60$ (s, 1H), 7.76 (d, $J = 15.9$ Hz, 1H), 7.52 (d, $J = 8.6$ Hz, 2H), 6.93 (d, $J = 8.5$ Hz, 2H), 6.33 (d, $J = 15.9$ Hz, 1H), 3.86 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3): $\delta = 172.3$, 161.7, 146.7, 130.1, 126.8, 114.6, 114.4, 55.4.

(E)-Methyl 3-*p*-tolylacrylate (Table 2, 6n)⁵



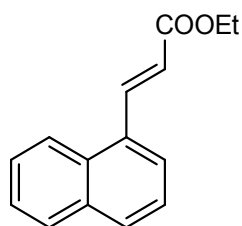
White solid; m.p. 56-58 °C; IR (KBr): $\nu = 1711.5 \text{ cm}^{-1}$; $^1\text{H NMR}$ (300 MHz, CDCl_3): $\delta = 7.68$ (d, $J = 16.0$ Hz, 1H), 7.42 (d, $J = 8.0$ Hz, 2H), 7.18 (d, $J = 7.9$ Hz, 2H), 6.41 (d, $J = 16.0$ Hz, 1H), 3.80 (s, 3H), 2.37 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3): $\delta = 167.6, 144.8, 140.7, 131.7, 129.6, 128.1, 116.7, 51.6, 21.4$.

(E)-Methyl 2-methyl-3-phenylacrylate (Table 2, 6p)⁶



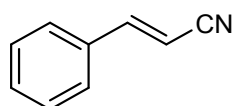
Light yellow oil; IR (KBr): $\nu = 1714.4 \text{ cm}^{-1}$; $^1\text{H NMR}$ (300 MHz, CDCl_3): $\delta = 7.71$ (s, 1H), 7.41 (d, $J = 4.2$ Hz, 2H), 7.28-7.37 (m, 3H), 3.84 (s, 3H), 2.14 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3): $\delta = 169.2, 135.9, 130.7, 129.6, 129.2, 128.5, 128.4, 52.1, 14.1$.

(E)-Ethyl 3-(naphthalen-5-yl)acrylate (Table 2, 6q)⁶



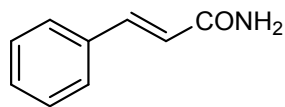
Light yellow oil; IR (KBr): $\nu = 1711.4 \text{ cm}^{-1}$; $^1\text{H NMR}$ (300 MHz, CDCl_3): $\delta = 8.56$ (d, $J = 15.8$ Hz, 1H), 8.21 (d, $J = 8.2$ Hz, 1H), 7.86-7.90 (m, 2H); 7.75 (d, $J = 7.2$ Hz, 1H), 7.45-7.61 (m, 3H), 6.55 (d, $J = 15.8$ Hz, 1H), 4.33 (q, $J = 7.1$ Hz, 2H), 1.40 (t, $J = 7.1$ Hz, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3): $\delta = 166.9, 141.6, 133.7, 131.8, 131.4, 130.5, 128.7, 126.8, 126.2, 125.5, 124.99, 123.4, 120.9, 60.6, 14.4$.

(E)-Cinnamionitrile (Table 2, 6r)²



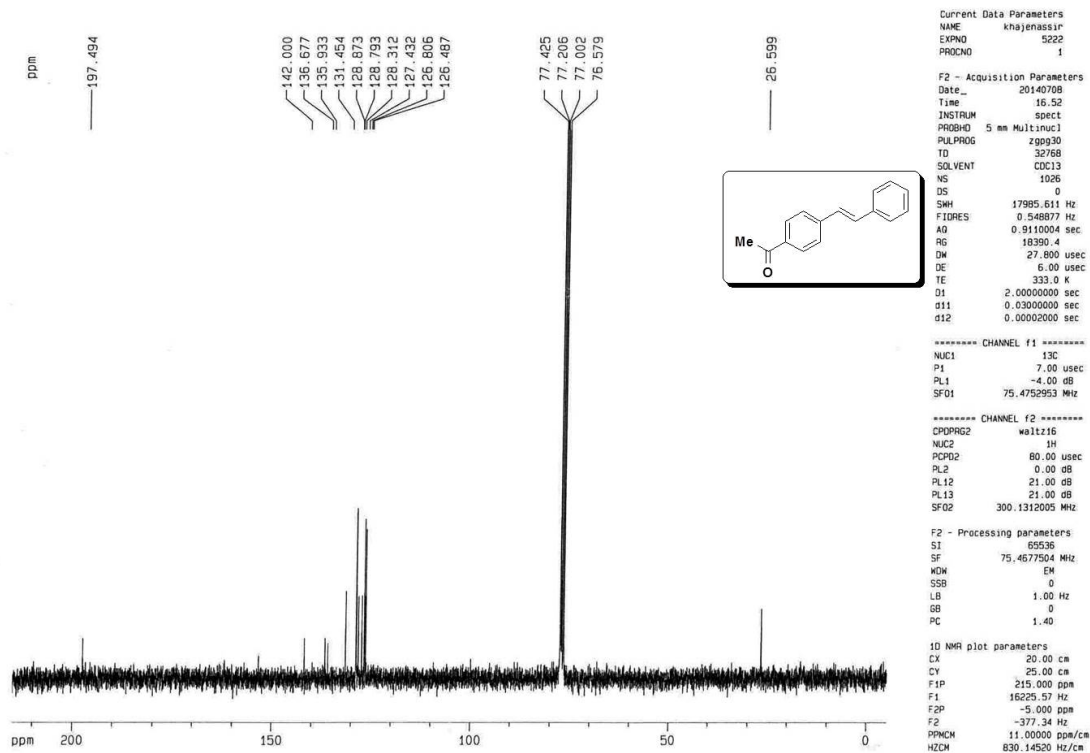
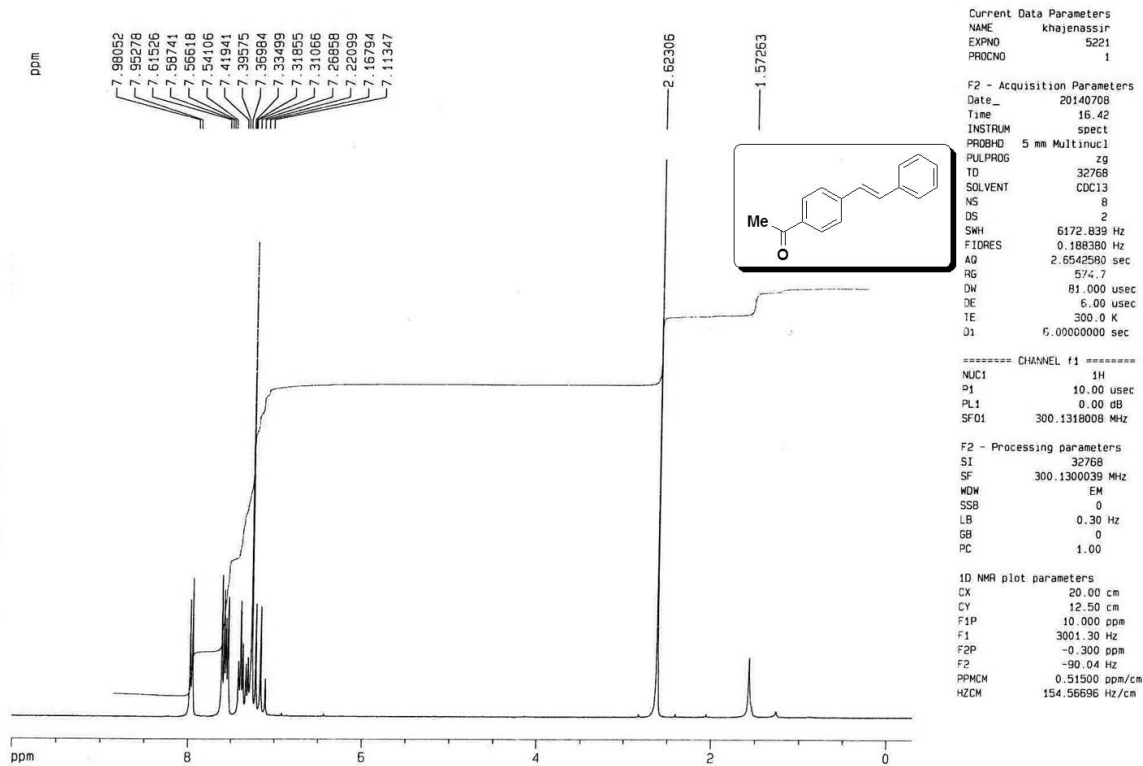
Light yellow oil; IR (neat): $\nu = 2217.7 \text{ cm}^{-1}$; $^1\text{H NMR}$ (300 MHz, CDCl_3): $\delta = 7.81$ -7.83 (m, 1H), 7.43-7.44 (m, 4H), 7.39 (d, $J = 16.9$ Hz, 1H), 5.88 (d, $J = 16.6$ Hz, 1H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3): $\delta = 150.6, 131.2, 129.1, 128.9, 127.4, 118.2, 96.3$.

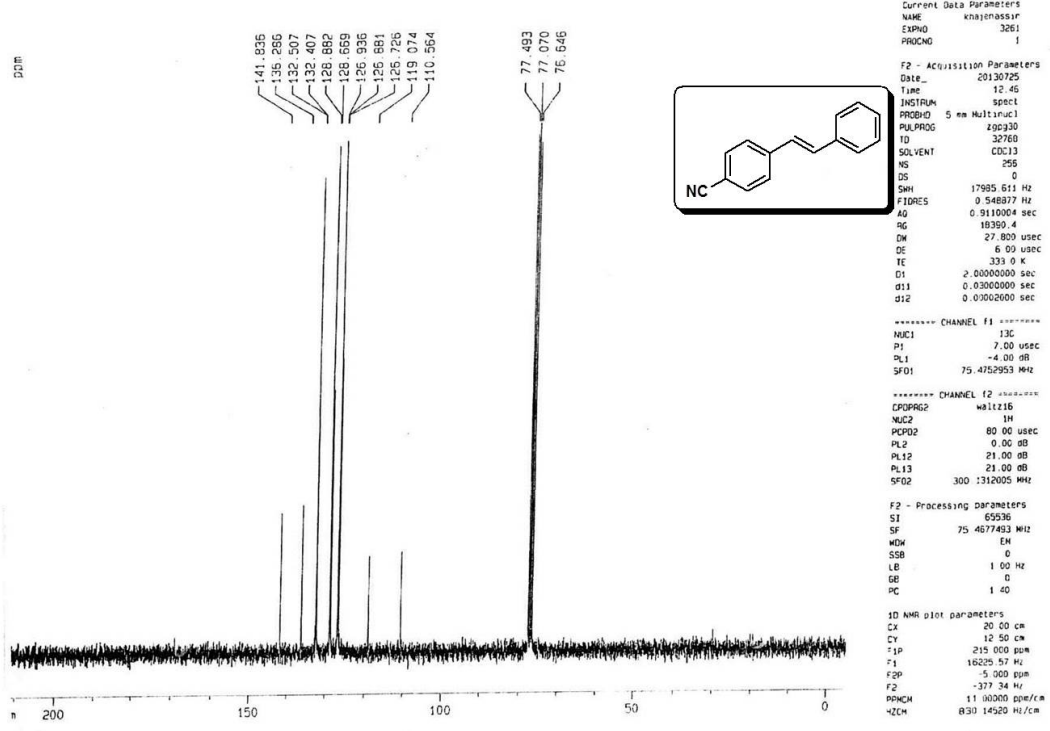
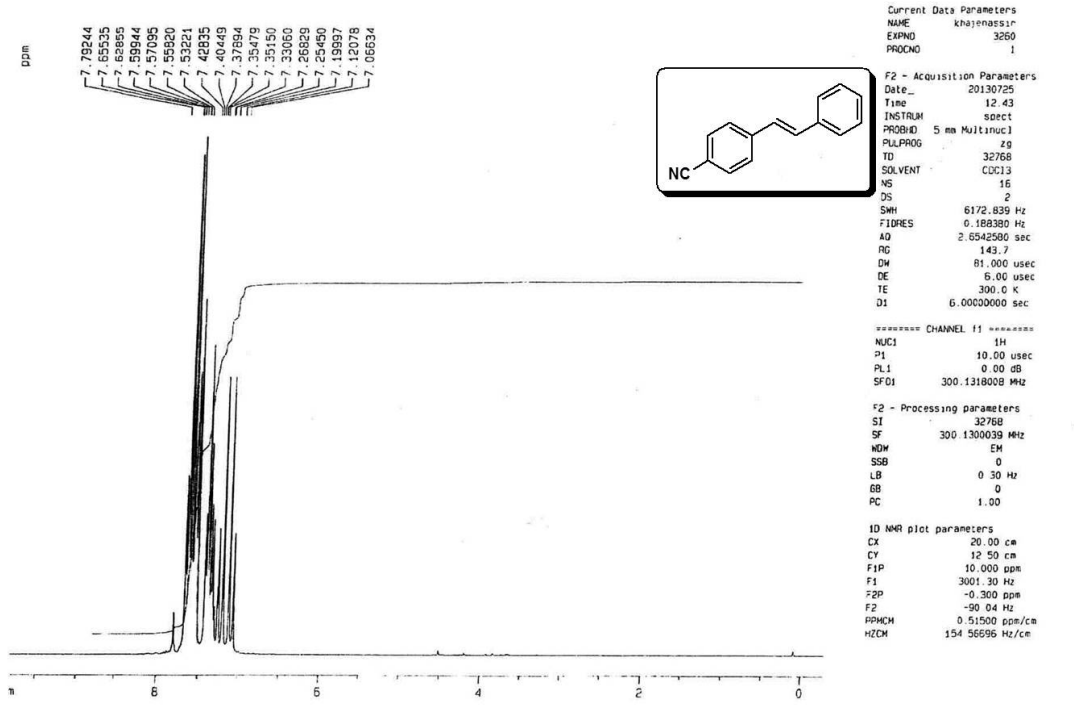
(E)-Cinnamamide (Table 2, 6s)²

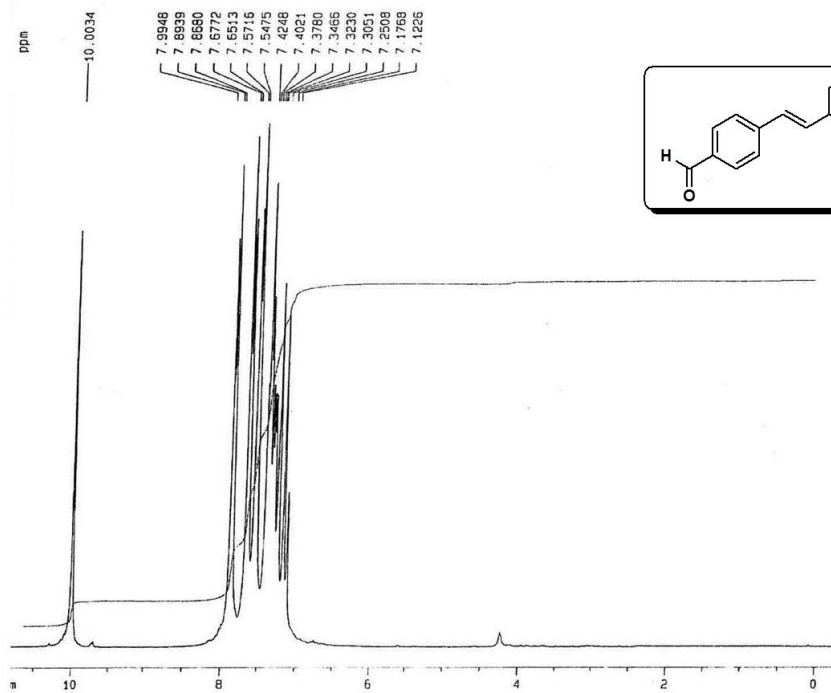


Pale-yellow solid; mp 147–149 °C; IR (KBr): $\nu = 3575.4, 3170.9, 1662.4 \text{ cm}^{-1}$; $^1\text{H NMR}$ (300 MHz, CDCl_3): $\delta = 7.64$ (d, $J = 15.7 \text{ Hz}$, 1H), 7.49 (s, 2H), 7.36 (s, 3H), 6.49 (d, $J = 15.7 \text{ Hz}$, 1H), 6.15 (brs, 1H), 5.97 (brs, 1H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3): $\delta = 168.0, 142.4, 134.5, 129.9, 128.8, 127.9, 119.6$.

3. Copy of ¹H- and ¹³C-NMR spectra of products Mizoroki-Heck reaction







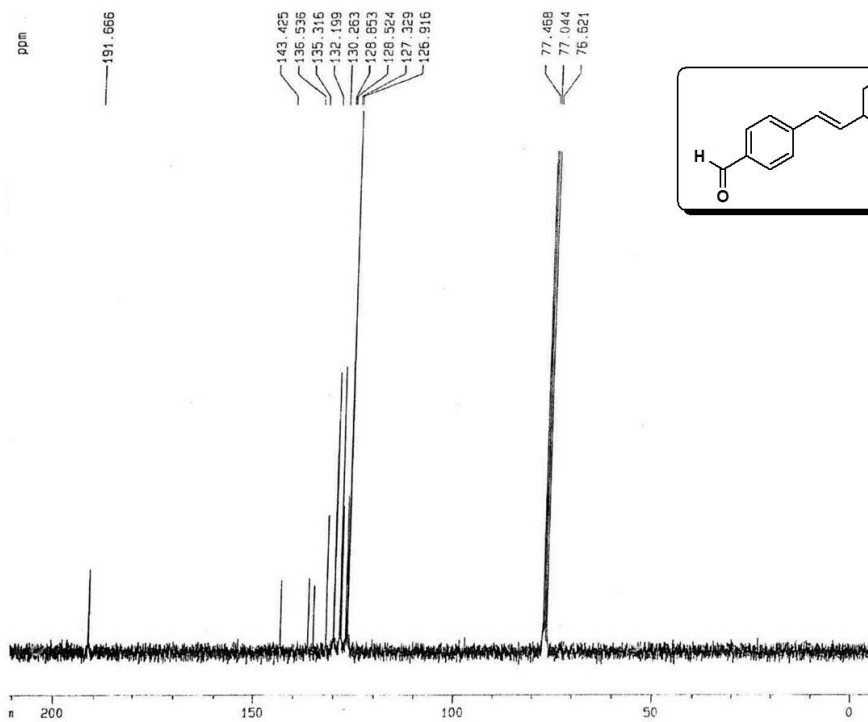
Current Data Parameters
 NAME khajenassin
 EXPNO 3262
 PROCNO 1

F2 - Acquisition Parameters
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 Time 13.03
 INSTRUM spect
 PROBHD 5 mm Multinuc1
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6172.835 Hz
 FIDRES 0.188380 Hz
 AQ 2.6542580 sec
 RG 181
 DM 81.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 6.00000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 10.00 usec
 PL1 0.00 dB
 SFO1 300.1316008 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300039 MHz
 MDW EH
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 1.000 ppm
 F1 3301.43 Hz
 F2P -0.300 ppm
 F2 -90.04 Hz
 PRMCH 0.56500 ppm/cm
 HZCM 169.57344 Hz/cm



Current Data Parameters
 NAME khajenassin
 EXPNO 3263
 PROCNO 1

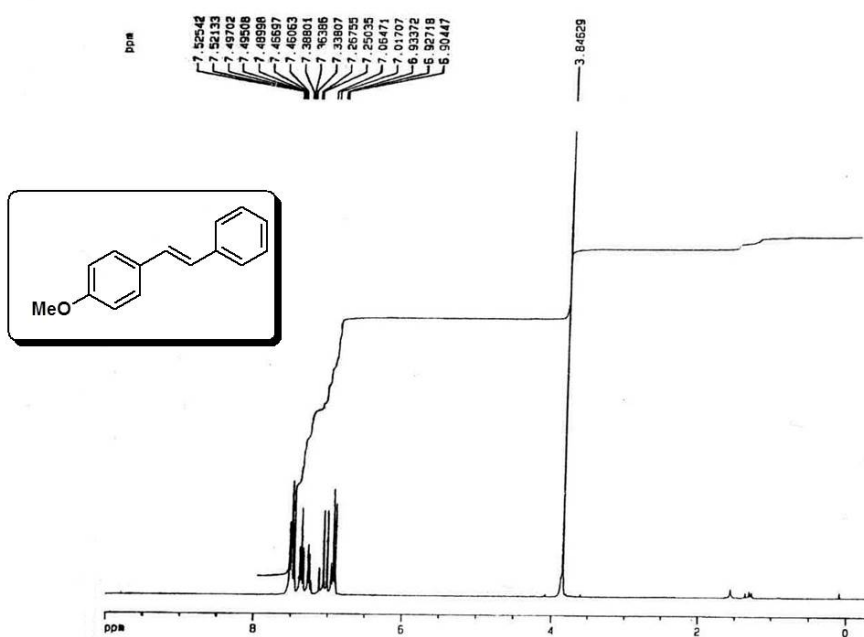
F2 - Acquisition Parameters
 Date_ 20130725
 Time 13.19
 INSTRUM spect
 PROBHD 5 mm Multinuc1
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 600
 DS 0
 SWH 17985.511 Hz
 FIDRES 0.546877 Hz
 AQ 0.8110004 sec
 RG 18350.4
 DM 27.800 usec
 DE 6.00 usec
 TE 333.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 7.00 usec
 PL1 -4.00 dB
 SFO1 75.4752953 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 21.00 dB
 PL13 21.00 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 65536
 SF 75.4677463 MHz
 MDW EH
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 215.000 ppm
 F1 16225.57 Hz
 F2P -5.000 ppm
 F2 -377.34 Hz
 PRMCH 11.00000 ppm/cm
 HZCM 830.14520 Hz/cm



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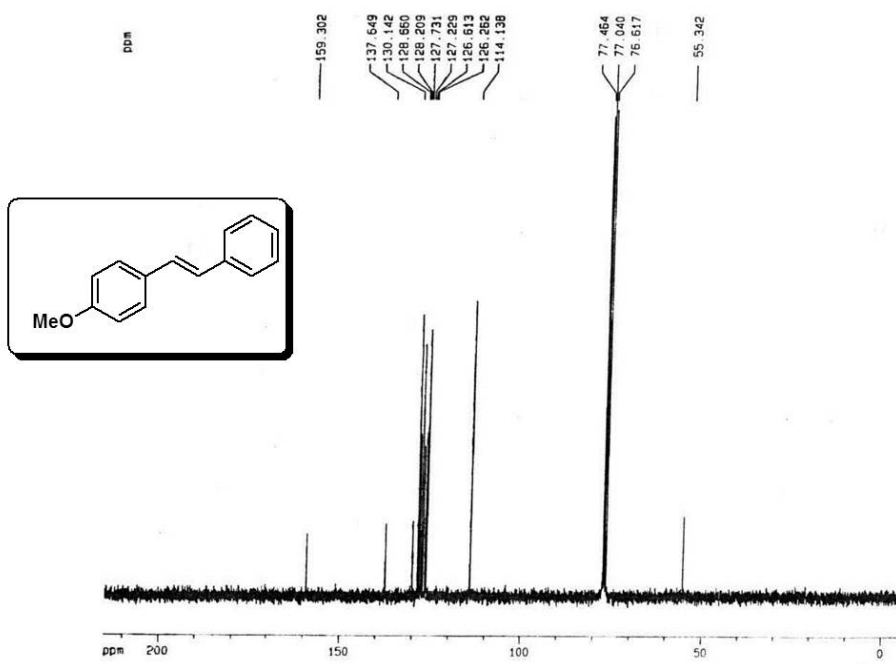
Current Data Parameters
NAME      khajenassir
EXPNO    3245
PROCNO   1

F2 - Acquisition Parameters
Date_    20130702
Time     20.23
INSTRUM  spect
PROBHD   5 mm Multinucl
PULPROG  zg
TD        32768
SOLVENT  CDCl3
NS        16
DS        2
SMH       6172.839 Hz
FIDRES   0.188280 Hz
AQ        2.6542800 sec
RG        228.1
DM        81.000 usec
DE        6.00 usec
TE        300.0 K
D1        6.0000000 sec

----- CHANNEL f1 -----
NUC1      1H
P1        10.00 usec
PL1       0.00 dB
SFO1     300.1318008 MHz

F2 - Processing parameters
SI        32768
SF        300.1300299 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00

1D NMR plot parameters
CX        20.00 cm
CY        12.50 cm
F1P       10.000 ppm
F1        3001.30 Hz
F2P       -0.300 ppm
F2        -90.04 Hz
PPICH    0.51500 ppm/cm
HZCH     154.56696 Hz/cm
    
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Current Data Parameters
NAME      khajenassir
EXPNO    3246
PROCNO   1

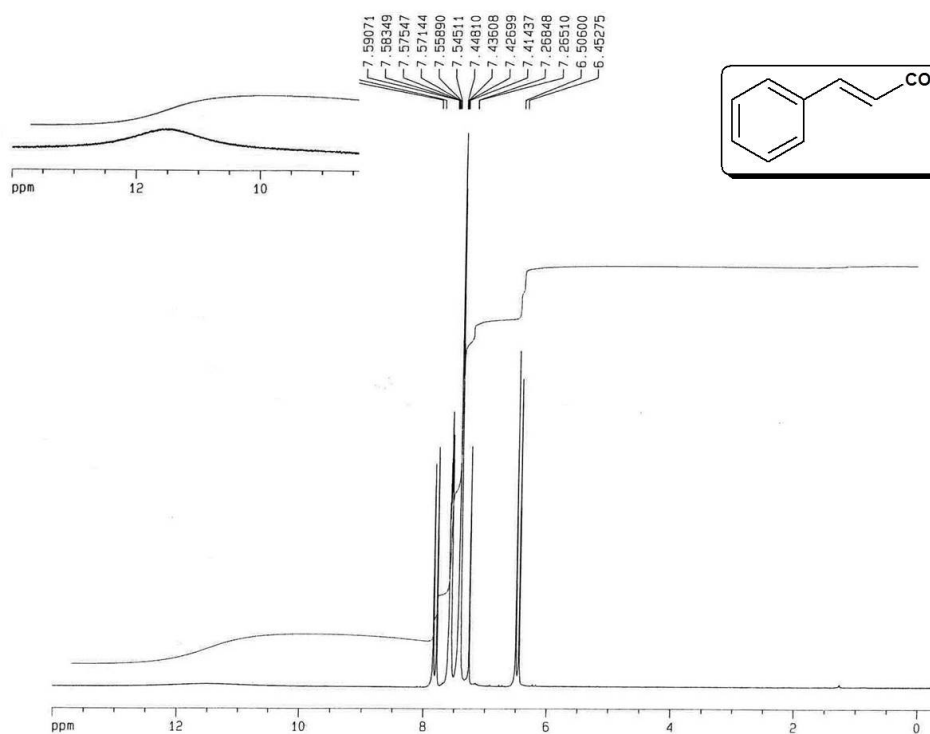
F2 - Acquisition Parameters
Date_    20130702
Time     20.28
INSTRUM  spect
PROBHD   5 mm Multinucl
PULPROG  zgpg30
TD        32768
SOLVENT  CDCl3
NS        540
DS        0
SMH       17985.611 Hz
FIDRES   0.548877 Hz
AQ        0.9110004 sec
RG        18390.4
DM        27.800 usec
DE        6.00 usec
TE        333.0 K
D1        2.0000000 sec
d11       0.0300000 sec
d12       0.00002000 sec

----- CHANNEL f1 -----
NUC1      13C
P1        7.00 usec
PL1       -4.00 dB
SFO1     75.4732963 MHz

----- CHANNEL f2 -----
CPDPRG2  waltz16
NUC2      1H
PCPD2    80.00 usec
PL2       0.00 dB
PL12     21.00 dB
PL13     21.00 dB
SFO2     300.1312005 MHz

F2 - Processing parameters
SI        65536
SF        75.4677493 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40

1D NMR plot parameters
CX        20.00 cm
CY        12.50 cm
F1P       215.000 ppm
F1        18229.57 Hz
F2P       -5.000 ppm
F2        -377.34 Hz
PPICH    11.00000 ppm/cm
HZCH     838.14520 Hz/cm
    
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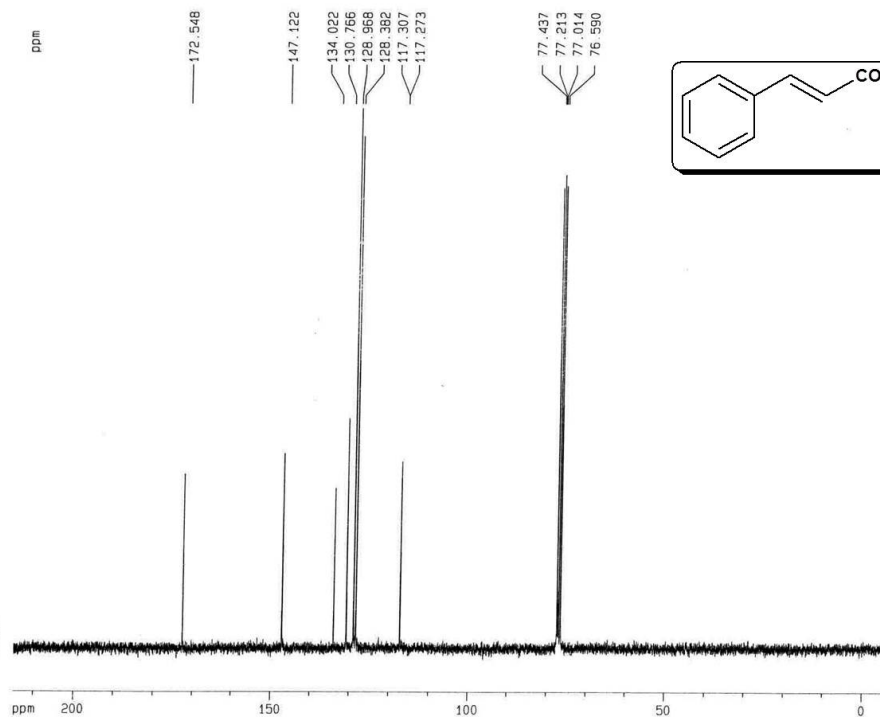
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 NAME khajenassir
 EXPNO 5219
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20140707
 Time 18.04
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.188380 Hz
 AQ 2.6542580 sec
 RG 228.1
 DW 81.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 6.0000000 sec

CHANNEL f1
 NUC1 1H
 P1 10.00 usec
 PL1 0.00 dB
 SFO1 300.1318008 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300039 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 14.000 ppm
 F1 4201.82 Hz
 F2P -0.300 ppm
 F2 -90.04 Hz
 PPMCM 0.71500 ppm/cm
 HZCM 214.59296 Hz/cm



Current Data Parameters
 NAME khajenassir
 EXPNO 5220
 PROCNO 1

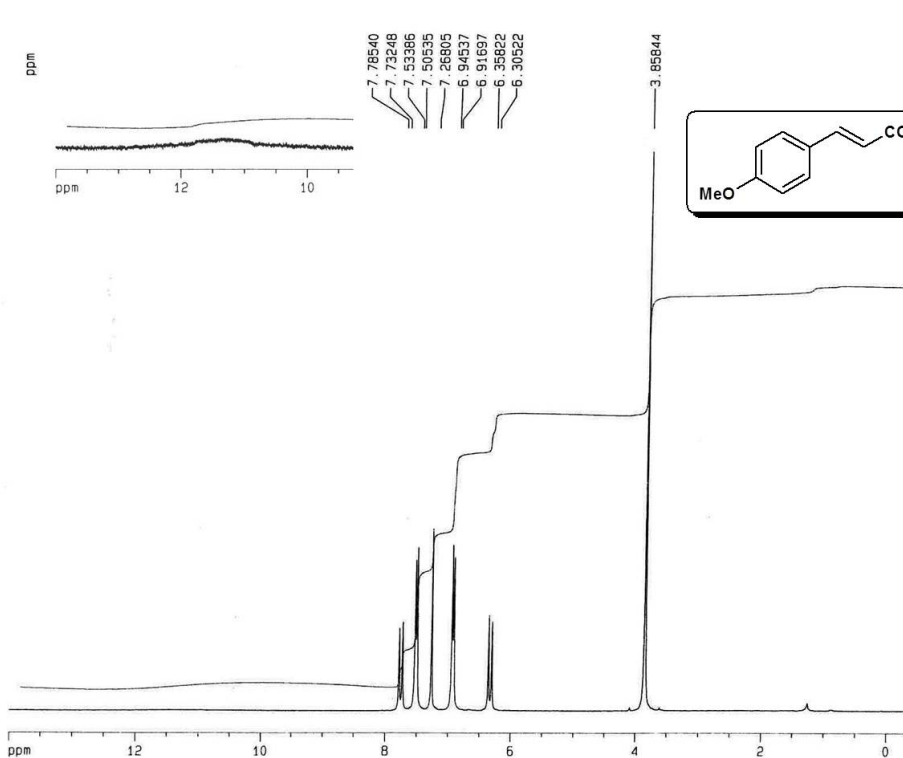
F2 - Acquisition Parameters
 Date_ 20140707
 Time 18.07
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 835
 DS 0
 SWH 17985.611 Hz
 FIDRES 0.548877 Hz
 AQ 0.9110004 sec
 RG 18390.4
 DW 27.800 usec
 DE 6.00 usec
 TE 333.0 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 d12 0.0000200 sec

CHANNEL f1
 NUC1 13C
 P1 7.00 usec
 PL1 -4.00 dB
 SFO1 75.4752953 MHz

CHANNEL f2
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 21.00 dB
 PL13 21.00 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 65536
 SF 75.4677504 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 215.000 ppm
 F1 16225.57 Hz
 F2P -5.000 ppm
 F2 -377.34 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 830.14520 Hz/cm



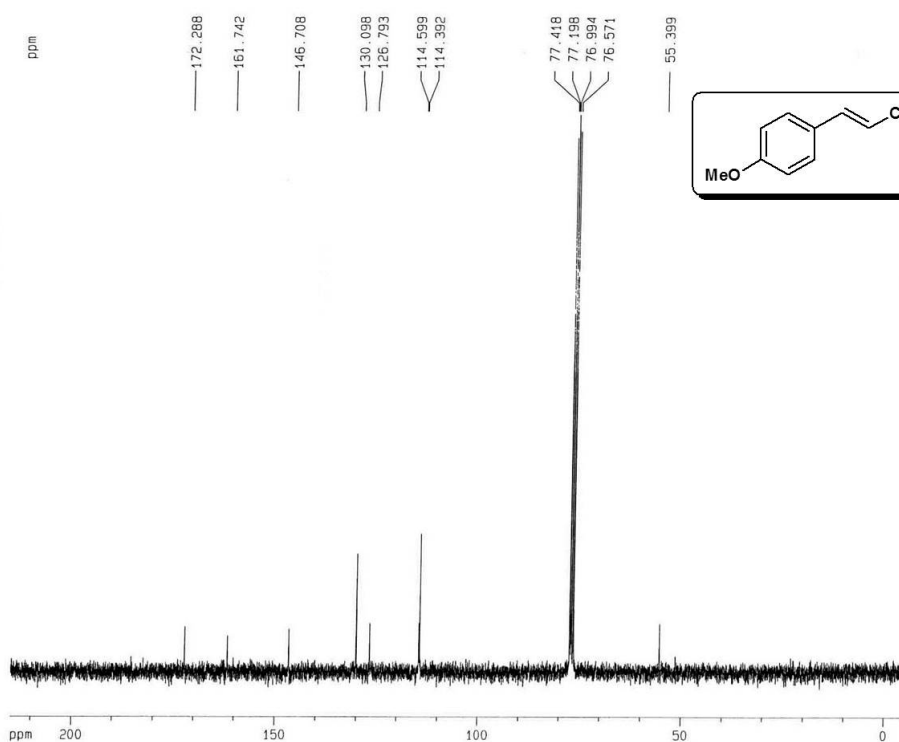
Current Data Parameters
 NAME khajenassir
 EXPNO 5225
 PROCNO 1

F2 - Acquisition Parameters
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 Time 12.20
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.188380 Hz
 AQ 2.6542580 sec
 RG 406.4
 DW 81.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 6.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.00 usec
 PL1 0.00 dB
 SFO1 300.1318008 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300039 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 14.000 ppm
 F1 4201.82 Hz
 F2P -0.300 ppm
 F2 -90.04 Hz
 PPMCM 0.71500 ppm/cm
 HZCM 214.59296 Hz/cm



Current Data Parameters
 NAME khajenassir
 EXPNO 5226
 PROCNO 1

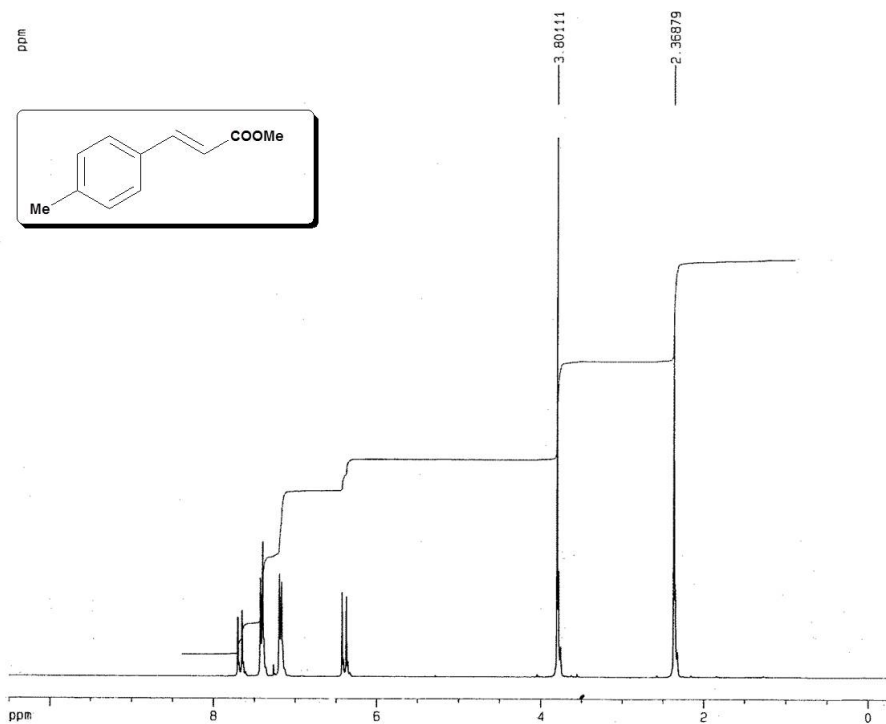
F2 - Acquisition Parameters
 Date_ 20140709
 Time 12.27
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 770
 DS 0
 SWH 17985.611 Hz
 FIDRES 0.548877 Hz
 AQ 0.9110004 sec
 RG 18390.4
 DW 27.800 usec
 DE 6.00 usec
 TE 333.0 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 c12 0.0002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 7.00 usec
 PL1 -4.00 dB
 SFO1 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waitz15
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 21.00 dB
 PL13 21.00 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 65536
 SF 75.4677504 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 215.000 ppm
 F1 16225.57 Hz
 F2P -5.000 ppm
 F2 -377.34 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 830.14520 Hz/cm



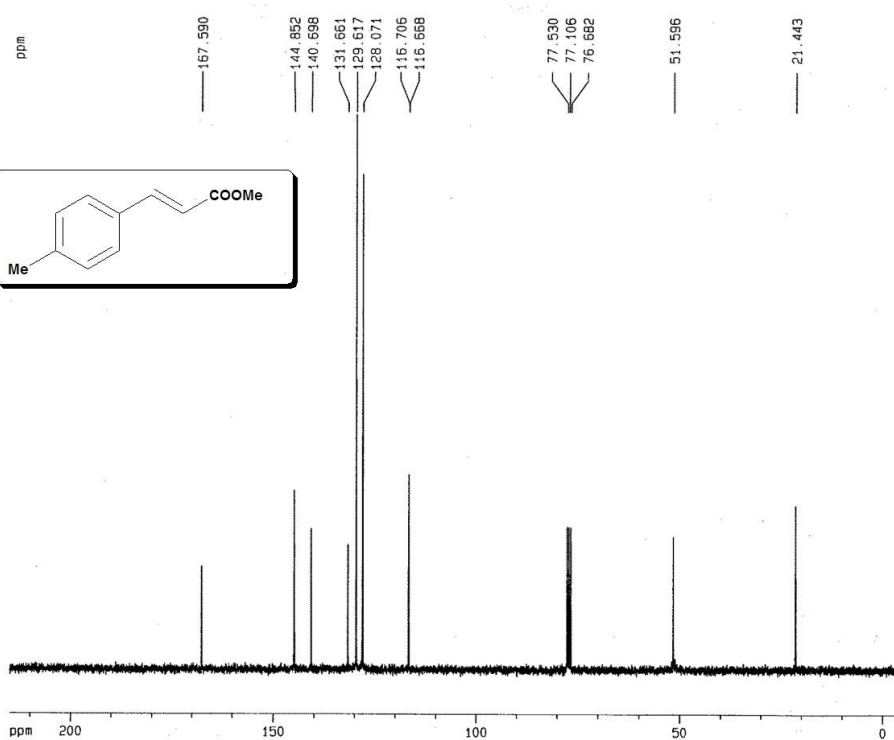
Current Data Parameters
 NAME khajenassin
 EXPNO 5203
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20140310
 Time 18.08
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 2
 SMH 6172.839 Hz
 FIDRES 0.188380 Hz
 AQ 2.6542960 sec
 RG 40.3
 DM 81.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 6.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.00 usec
 PL1 0.00 dB
 SFO1 300.1318008 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300039 MHz
 WDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 10.500 ppm
 F1 3151.36 Hz
 F2P -0.300 ppm
 F2 -90.04 Hz
 PPMCM 0.54000 ppm/cm
 HZCM 162.07021 Hz/cm



13C Data Parameters
 khajenassin
 5202
 1

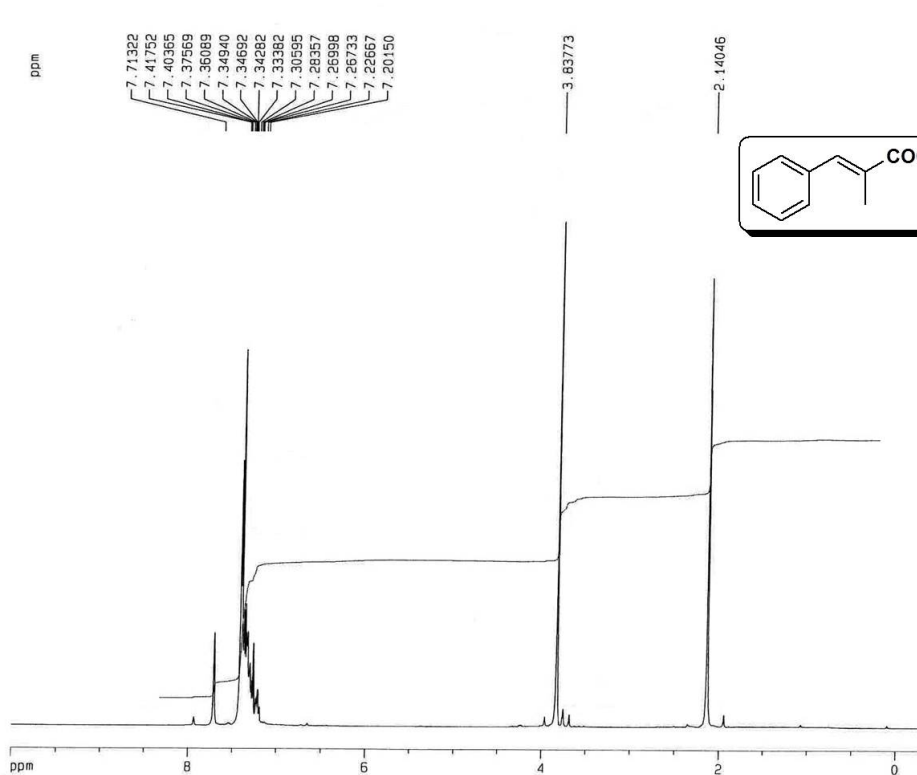
F2 - Acquisition Parameters
 Date_ 20140310
 Time 18.07
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 135
 DS 0
 SMH 17985.611 Hz
 FIDRES 0.548877 Hz
 AQ 0.9110004 sec
 RG 3251
 DM 27.800 usec
 DE 6.00 usec
 TE 333.0 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 d12 0.0000200 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 7.00 usec
 PL1 -4.00 dB
 SFO1 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 21.00 dB
 PL13 21.00 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 65536
 SF 75.4677493 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 215.000 ppm
 F1 16225.57 Hz
 F2P -5.000 ppm
 F2 -377.34 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 830.14520 Hz/cm



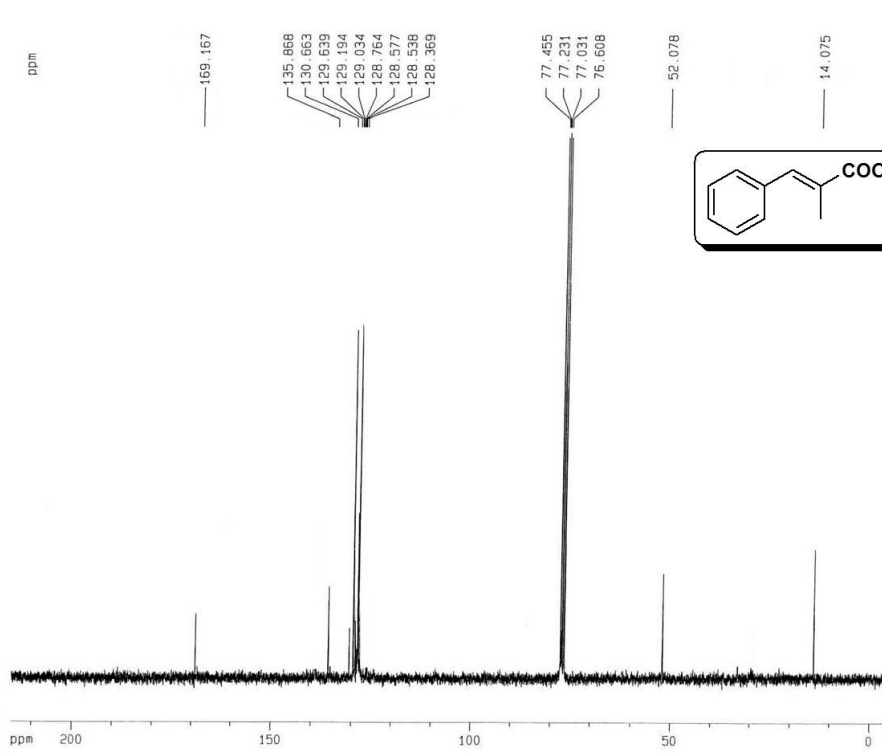
Current Data Parameters
 NAME khajenassir
 EXPNO 5217
 PROCNO 1

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 Time 15.13
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.188380 Hz
 AQ 2.6542580 sec
 RG 90.5
 DW 81.000 usec
 DE 5.00 usec
 TE 300.0 K
 D1 6.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.00 usec
 PL1 0.00 dB
 SF01 300.1318008 MHz

F2 - Processing parameters
 S1 32768
 SF 300.130039 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 10.000 ppm
 F1 3001.30 Hz
 F2P -0.300 ppm
 F2 -90.04 Hz
 PPMCM 0.51500 ppm/cm
 HZCM 154.56696 Hz/cm



t Data Parameters
 khajenassir
 PROCNO 5218

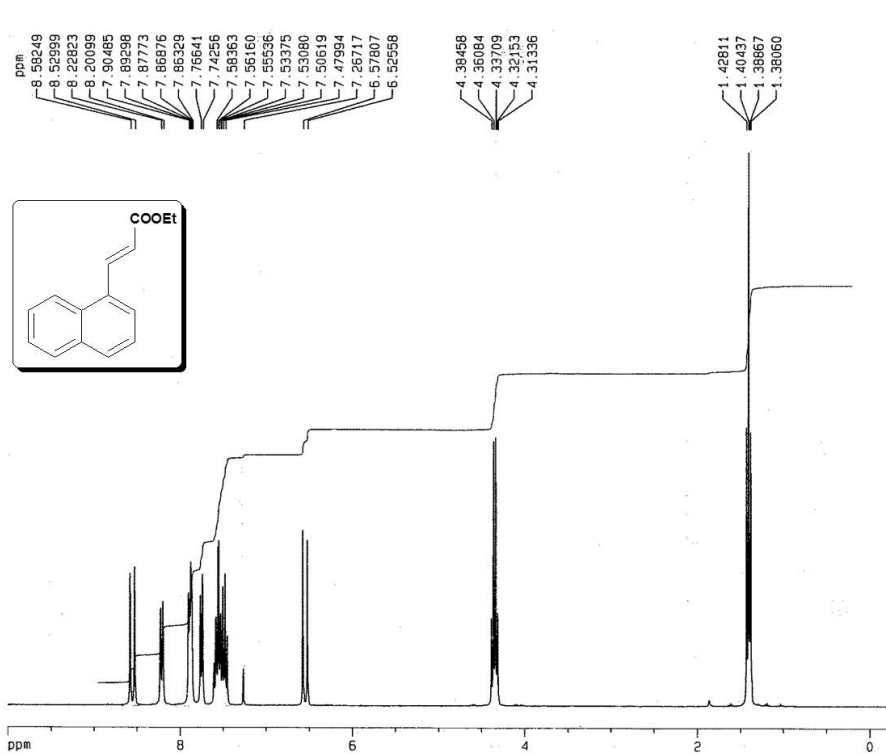
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 Time 15.25
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 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 705
 DS 0
 SWH 17985.611 Hz
 FIDRES 0.548877 Hz
 AQ 0.9110004 sec
 RG 18390.4
 DW 27.800 usec
 DE 6.00 usec
 TE 333.0 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 d12 0.0000200 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 7.00 usec
 PL1 -4.00 dB
 SF01 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 21.00 dB
 PL13 21.00 dB
 SF02 300.1312005 MHz

F2 - Processing parameters
 S1 65536
 SF 75.4677504 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 215.000 ppm
 F1 16225.57 Hz
 F2P -5.000 ppm
 F2 -377.34 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 830.14520 Hz/cm



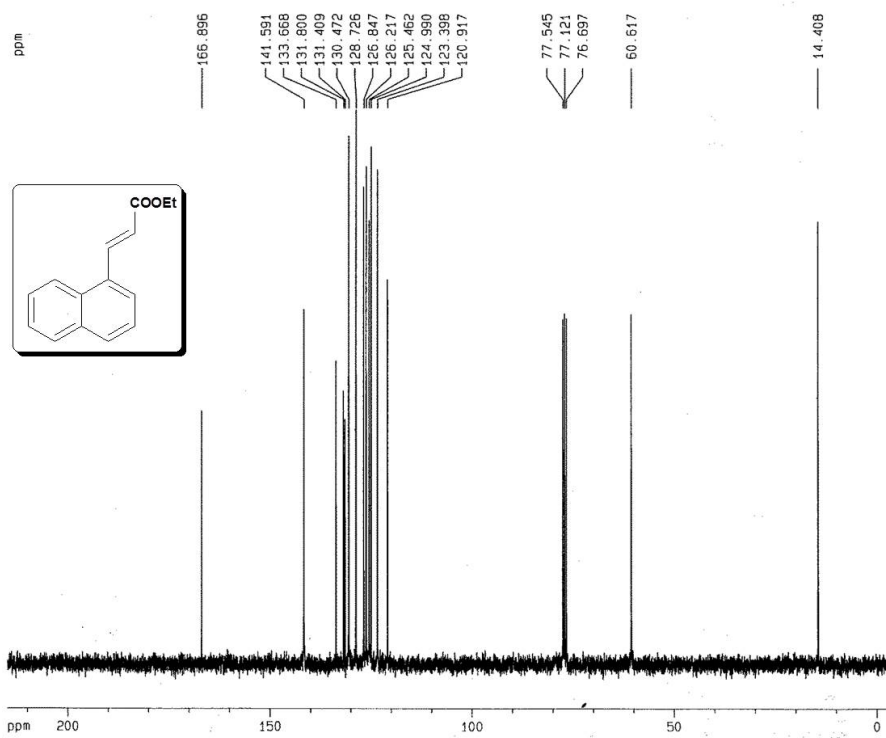
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 NAME khajenassir
 EXPNO 214
 PROCNO 1

F2 - Acquisition Parameters
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 Time 12.31
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.188380 Hz
 AQ 2.6542580 sec
 RG 40.3
 DW 81.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 6.00000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 10.00 usec
 PL1 0.00 dB
 SFO1 300.1318008 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300039 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 10,000 ppm
 F1 3001.30 Hz
 F2P -0.300 ppm
 F2 -90.04 Hz
 PPMCH 0.51500 ppm/cm
 HZCM 154.56696 Hz/cm



Current Data Parameters
 NAME khajenassir
 EXPNO 215
 PROCNO 1

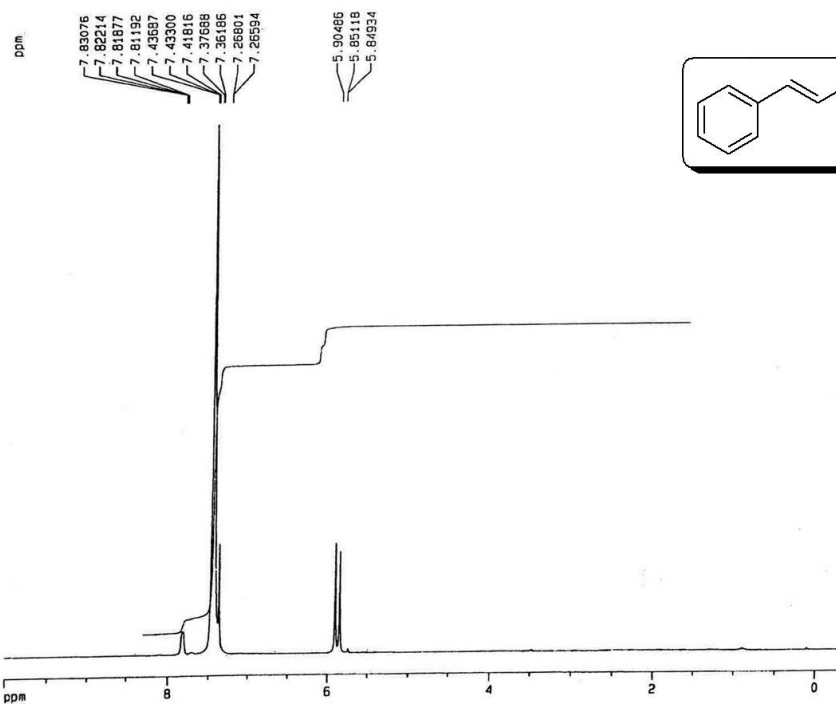
F2 - Acquisition Parameters
 Date_ 20140515
 Time 12.37
 INSTRUM spect
 PROBHD 5 mm Multinucl
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 128
 DS 0
 SWH 17985.611 Hz
 FIDRES 0.548877 Hz
 AQ 0.9110004 sec
 RG 20542.5
 DW 27.800 usec
 DE 6.00 usec
 TE 333.0 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 d12 0.00002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 7.00 usec
 PL1 -4.00 dB
 SFO1 75.4752953 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 21.00 dB
 PL13 21.00 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 65536
 SF 75.4677504 MHz
 WDW EN
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 215,000 ppm
 F1 16225.57 Hz
 F2P -5.000 ppm
 F2 -377.34 Hz
 PPMCH 11.00000 ppm/cm
 HZCM 830.14520 Hz/cm



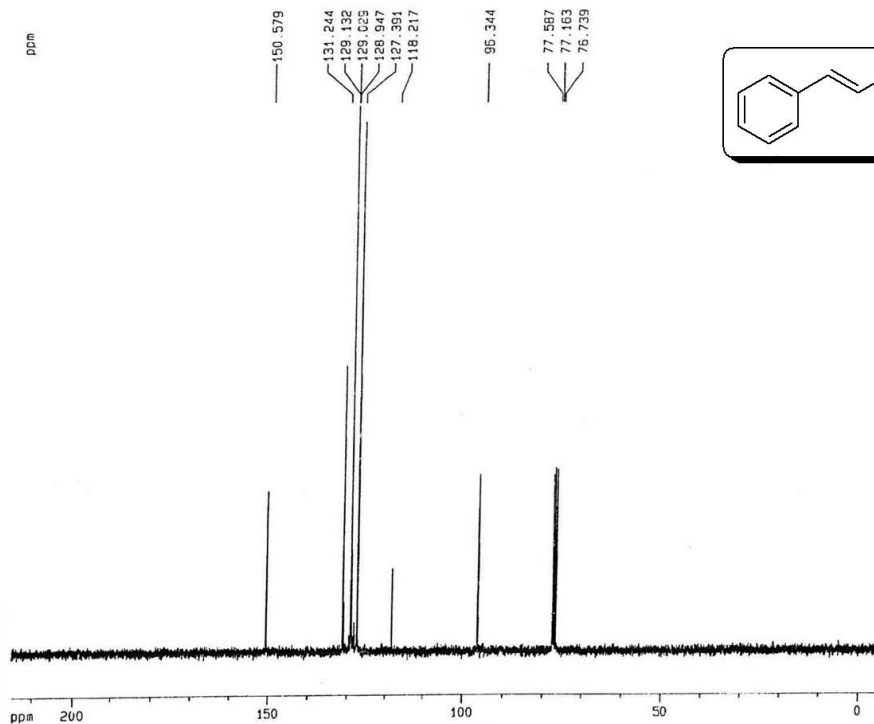
Current Data Parameters
 NAME khajenassir
 EXPNO 212
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20140515
 Time 12.17
 INSTRUM spect
 PROBHD 5 mm Multinuc1
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 2
 SMH 5172.839 Hz
 FIDRES 0.188380 Hz
 AQ 2.654260 sec
 RG 45.3
 DW 81.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 6.0000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 10.00 usec
 PL1 0.00 dB
 SFO1 300.1318008 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300039 MHz
 WDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 10.000 ppm
 F1 3001.30 Hz
 F2P -0.300 ppm
 F2 -90.04 Hz
 PPMCM 0.51500 ppm/cm
 HZCM 154.56696 Hz/cm



Current Data Parameters
 NAME khajenassir
 EXPNO 213
 PROCNO 1

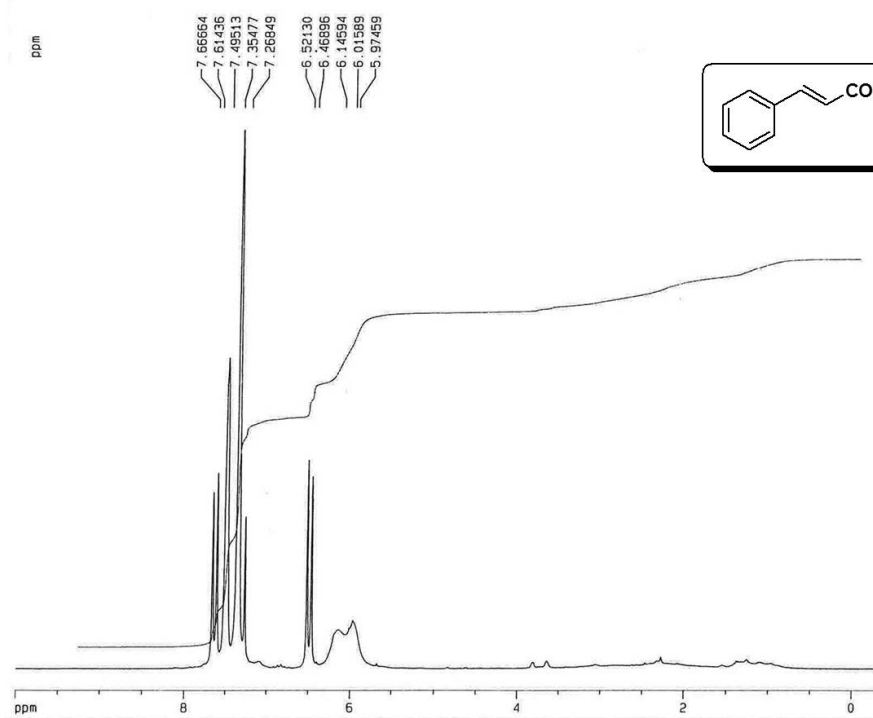
F2 - Acquisition Parameters
 Date_ 20140515
 Time 12.19
 INSTRUM spect
 PROBHD 5 mm Multinuc1
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 128
 DS 0
 SMH 17995.611 Hz
 FIDRES 0.548877 Hz
 AQ 0.9110004 sec
 RG 20642.5
 DW 27.800 usec
 DE 8.00 usec
 TE 333.0 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 d12 0.0000200 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 7.00 usec
 PL1 -4.00 dB
 SFO1 75.4752953 MHz

***** CHANNEL f2 *****
 CHPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 21.00 dB
 PL13 21.00 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 65536
 SF 75.4577504 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 215.000 ppm
 F1 16225.57 Hz
 F2P -5.000 ppm
 F2 -377.34 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 830.14520 Hz/cm



```

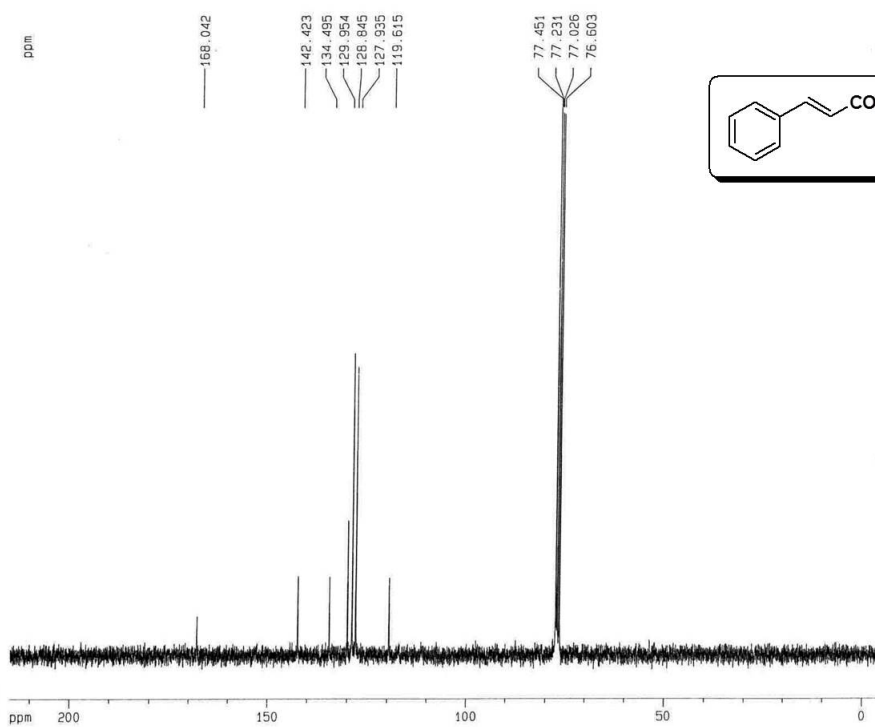
Current Data Parameters
NAME      khajenassir
EXPNO    5223
PROCNO   1

F2 - Acquisition Parameters
Date_    20140708
Time     18.42
INSTRUM  spect
PROBHD   5 mm Multinucl
PULPROG  zg
TD        32768
SOLVENT  CDCl3
NS        8
DS        2
SWH       6172.839 Hz
FIDRES   0.188380 Hz
AQ        2.6542580 sec
RG        161.3
DM        81.000 usec
DE        6.00 usec
TE        300.0 K
D1        6.0000000 sec

===== CHANNEL f1 =====
NUC1     1H
P1       10.00 usec
PL1      0.00 dB
SFO1     300.1318008 MHz

F2 - Processing parameters
SI        32768
SF        300.1300039 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00

1D NMR plot parameters
CX        20.00 cm
CY        12.50 cm
F1P       10.000 ppm
F1        3001.30 Hz
F2P       -0.300 ppm
F2        -90.04 Hz
PPMCH     0.51500 ppm/cm
HZCM      154.56696 Hz/cm
    
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```

Current Data Parameters
NAME      khajenassir
EXPNO    5224
PROCNO   1

F2 - Acquisition Parameters
Date_    20140708
Time     19.13
INSTRUM  spect
PROBHD   5 mm Multinucl
PULPROG  zgpg30
TD        32768
SOLVENT  CDCl3
NS        620
DS        0
SWH       17985.611 Hz
FIDRES   0.548877 Hz
AQ        0.9110004 sec
RG        18390.4
DM        27.800 usec
DE        6.00 usec
TE        333.0 K
D1        2.0000000 sec
d11      0.0300000 sec
d12      0.0000200 sec

===== CHANNEL f1 =====
NUC1     13C
P1       7.00 usec
PL1      -4.00 dB
SFO1     75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    80.00 usec
PL2      0.00 dB
PL12     21.00 dB
PL13     21.00 dB
SFO2     300.1312005 MHz

F2 - Processing parameters
SI        65536
SF        75.4677564 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40

1D NMR plot parameters
CX        20.00 cm
CY        12.50 cm
F1P       215.000 ppm
F1        16225.57 Hz
F2P       -5.000 ppm
F2        -377.34 Hz
PPMCH     11.00000 ppm/cm
HZCM      622.44220 Hz/cm
    
```

References

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