Supporting Information

Unsupported Nanoporous Gold Catalyst for Highly Selective Hydroamination of Alkynes

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1. General Information

^{1}H NMR spectra was recorded on a Bruker Ascend 500 M NMR spectrometer; $CDCl_3$ was used as a solvent, while tetramethylsilane (TMS) was used as an internal standard. The chemical shifts are reported in the ppm down field (δ) from TMS, and the coupling constants J are expressed in Hz. The peak patterns are labeled as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. Thin-layer chromatography (TLC) was carried out on SiO_2 (silica gel $60F_{254}$, Merck), and the spots were located with UV light, iodoplatinate reagent, or 1% aqueous $KMnO_4$. Flash chromatography was carried out on SiO_2 (silica gel 60, 200–300 mesh).

2. Preparation of AuNPore Catalyst

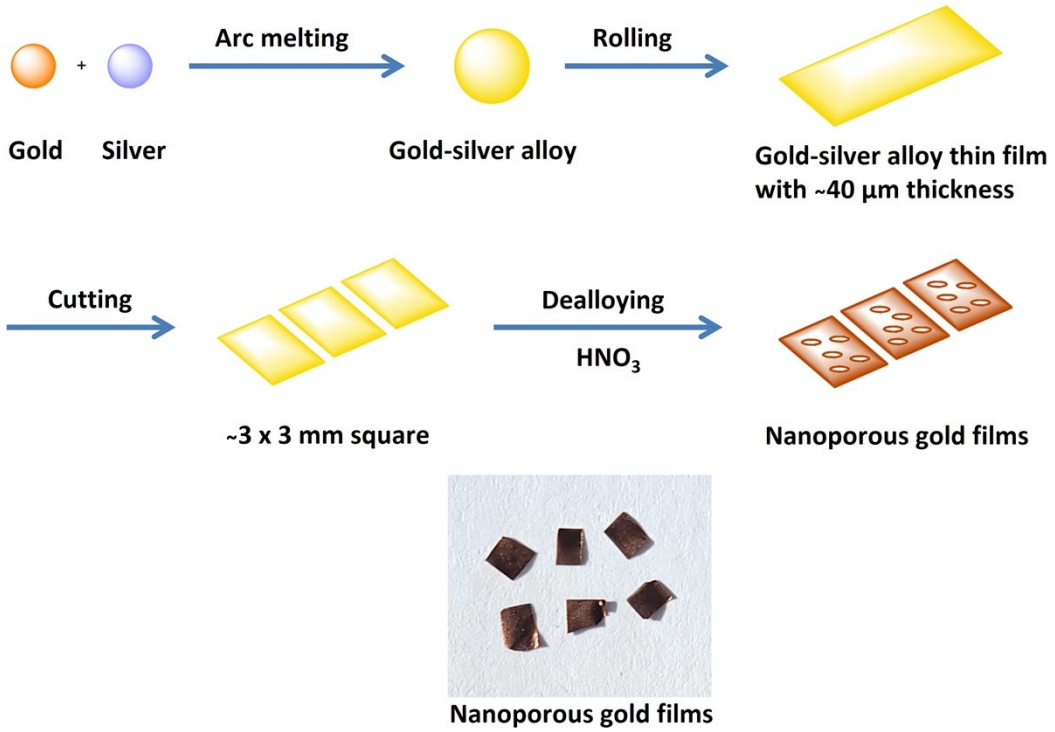


Figure S1. Schematic illustration of the fabrication process of nanoporous gold catalyst.

3. Characterization of Catalyst

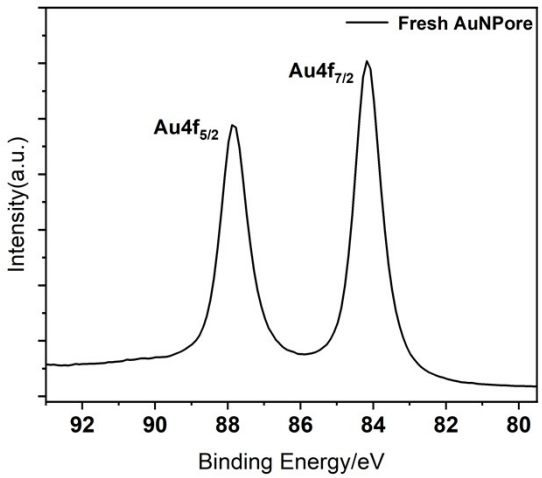


Figure S2. XPS spectra of Au 4f of fresh AuNPore.

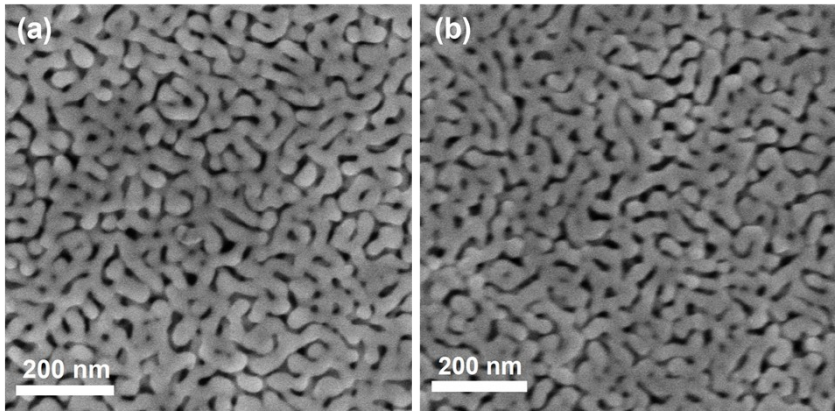


Figure S3. SEM images of (a) fresh AuNPore and (b) AuNPore after six run

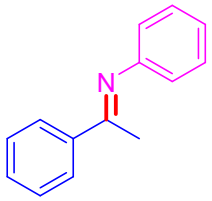
4. Representative Procedure for the AuNPore-Catalyzed One-Pot, Two-Step Synthesis of Secondary Amines

AuNPore (4.92 mg, 5 mol %), phenylacetylene (**1a**, 51.07 mg, 0.5 mmol), and phenylamine (**2a**, 55.55 mg, 0.6 mmol) were placed in a V-shaped vial reactor with a magnetic stir bar under N_2 atmosphere. The reaction mixture was stirred at 50 ºC for 24 h and cooled down to room temperature. Then, $PhMe_2SiH$ (102.20 mg, 0.75 mmol) and H_2O (18 mg, 1.0 mmol) were added directly to reaction mixture and stirring was continued at room temperature for 5 h. The AuNPore was recovered by filtration, followed by washing with acetone, and the residual was purified via silica gel chromatography (eluent: petroleum ether/ethyl acetate = 5/1) to afford secondary

amines (**4a**) as a colorless oil.

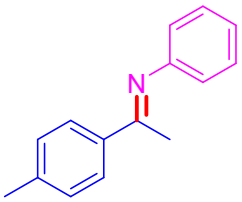
5. Characterization Data of Products

_N_,1-Diphenylethan-1-imine (3a)



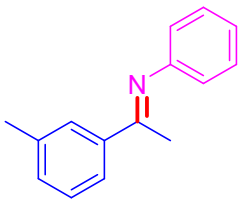
Yellow oil (91.8 mg, 94% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.02 (dd, J = 7.7, 2.0 Hz, 2H), 7.54–7.47 (m, 3H), 7.40 (t, J = 7.8 Hz, 2H), 7.13 (t, J = 7.4 Hz, 1H), 6.89–6.81 (m, 2H), 2.28 (s, 3H). NMR data are in agreement with those from the literature.[1]

_N_-Phenyl-1-(_p_-tolyl)ethan-1-imine (3b)



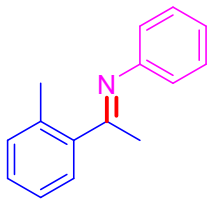
Yellow oil (84.8 mg, 81% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.87 (d, J = 8.2 Hz, 2H), 7.34 (t, J = 7.8 Hz, 2H), 7.25 (d, J = 8.2 Hz, 2H), 7.08 (t, J = 7.4 Hz, 1H), 6.82–6.77 (m, 2H), 2.41 (s, 3H), 2.21 (s, 3H). NMR data are in agreement with those from the literature.[1]

_N_-Phenyl-1-(_m_-tolyl)ethan-1-imine (3c)



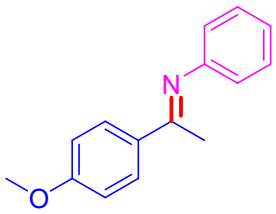
Yellow oil (87.9 mg, 84% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.82 (s, 1H), 7.72 (d, J = 8.0 Hz, 1H), 7.38–7.31 (m, 3H), 7.28 (d, J = 7.6 Hz, 1H), 7.12–7.05 (m, 1H), 6.83–6.76 (m, 2H), 2.42 (s, 3H), 2.22 (s, 3H). NMR data are in agreement with those from the literature.[1]

***N*-Phenyl-1-(*o*-tolyl)ethan-1-imine (3d)**



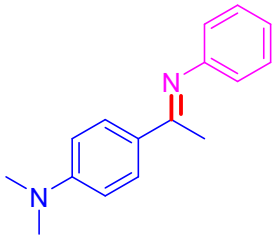
Yellow oil (85.8 mg, 82% yield, E/Z = 2:1). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.45–7.37 (m, 2H), 7.34-7.26 (m, 2H), 7.20–7.09 (m, 2H), 7.07–7.02 (m, 1H), 6.91–6.86 (m, 1.5H), 6.72–6.68 (m, 0.5H), 2.54 (s, 3H, major), 2.50 (s, 3H, minor), 2.19 (s, 3H, major), 2.14 (s, 3H, minor). NMR data are in agreement with those from the literature.[2]

1-(4-Methoxyphenyl)-*N*-phenylethan-1-imine (3e)



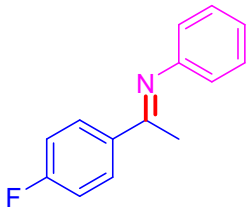
Yellow solid (90.1 mg, 80% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.94 (d, J = 9.0 Hz, 2H), 7.36–7.32 (m, 2H), 7.09–7.05 (m, 1H), 6.96–6.93 (m, 2H), 6.79–6.77 (m, 2H), 3.87 (s, 3H), 2.20 (s, 3H). NMR data are in agreement with those from the literature.[1]

***N*,*N*-Dimethyl-4-(1-(phenylimino)ethyl)aniline (3f)**



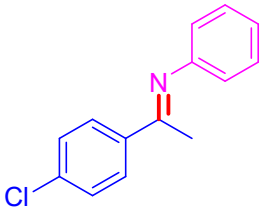
Brown solid (89.4 mg, 75% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.89 (d, J = 9.0 Hz, 2H), 7.32 (t, J = 8.0 Hz, 2H), 7.04 (t, J = 7.5 Hz, 1H), 6.79 (d, J = 7.0 Hz, 2H), 6.71 (d, J = 8.5 Hz, 2H), 3.02 (s, 6H), 2.17 (s, 3H). NMR data are in agreement with those from the literature.[1]

1-(4-Fluorophenyl)-*N*-phenylethan-1-imine (3g)



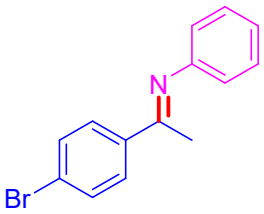
Yellow solid (93.8 mg, 88% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.01–7.95 (m, 2H), 7.35 (t, J = 7.9 Hz, 2H), 7.14–7.07 (m, 3H), 6.79 (d, J = 7.0 Hz, 2H), 2.22 (s, 3H). NMR data are in agreement with those from the literature.[1]

1-(4-Chlorophenyl)-*N*-phenylethan-1-imine (3h)



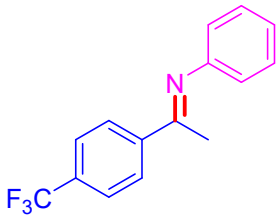
Yellow solid (97.6 mg, 85% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.92 (d, J = 8.5 Hz, 2H), 7.42 (d, J = 8.5 Hz, 2H), 7.36 (t, J = 7.5 Hz, 2H), 7.10 (t, J = 7.5 Hz, 1H), 6.79 (d, J = 7.5 Hz, 2H), 2.22 (s, 3H). NMR data are in agreement with those from the literature.[3]

1-(4-Bromophenyl)-*N*-phenylethan-1-imine (3i)



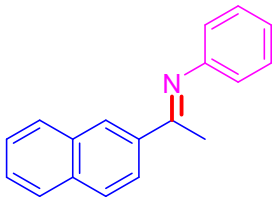
Yellow oil (115.1 mg, 84% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.85 (d, J = 8.6 Hz, 2H), 7.58 (d, J = 8.6 Hz, 2H), 7.35 (t, J = 7.8 Hz, 2H), 7.10 (t, J = 7.4 Hz, 1H), 6.78 (d, J = 7.0 Hz, 2H), 2.21 (s, 3H). NMR data are in agreement with those from the literature.[1]

***N*-Phenyl-1-(4-(trifluoromethyl)phenyl)ethan-1-imine (3j)**



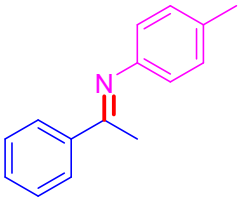
Yellow oil (118.5 mg, 90% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.09 (d, J = 8.1 Hz, 2H), 7.71 (d, J = 8.2 Hz, 2H), 7.37 (t, J = 7.9 Hz, 2H), 7.12 (t, J = 7.4 Hz, 1H), 6.84–6.76 (m, 2H), 2.27 (s, 3H). NMR data are in agreement with those from the literature.[1]

1-(Naphthalen-2-yl)-*N*-phenylethan-1-imine (3k)



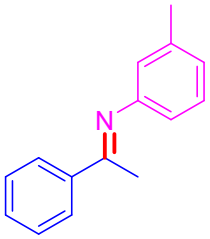
Yellow solid (104.3 mg, 85% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.35 (d, J = 1.8 Hz, 1H), 8.22 (dd, J = 8.6, 1.8 Hz, 1H), 7.99–7.83 (m, 3H), 7.60–7.48 (m, 2H), 7.43–7.32 (m, 2H), 7.17–7.04 (m, 1H), 6.90–6.75 (m, 2H), 2.36 (s, 3H). NMR data are in agreement with those from the literature.[3]

1-Phenyl-*N*-(p-tolyl)ethan-1-imine (3l)



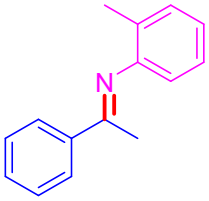
Yellow oil (97.3 mg, 93% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.98–7.95 (m, 2H), 7.46–7.43 (m, 3H), 7.16 (d, J = 8.0 Hz, 2H), 6.72–6.69 (m, 2H), 2.35 (s, 3H), 2.24 (s, 3H). NMR data are in agreement with those from the literature.[1]

1-Phenyl-*N*-(m-tolyl)ethan-1-imine (3m)



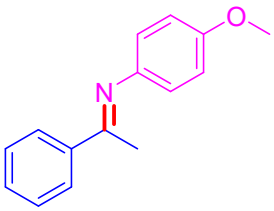
Yellow oil (95.2 mg, 91% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.99–7.93 (m, 2H), 7.48–7.42 (m, 3H), 7.24 (dd, J = 14.1, 6.4 Hz, 1H), 6.90 (d, J = 7.6 Hz, 1H), 6.65–6.57 (m, 2H), 2.36 (s, 3H), 2.24 (s, 3H). NMR data are in agreement with those from the literature.[4]

1-Phenyl-*N*-(o-tolyl)ethan-1-imine (3n)



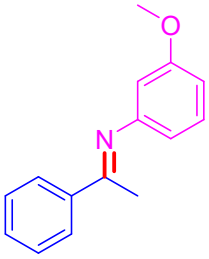
Yellow solid (94.2 mg, 90% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.04–7.88 (m, 2H), 7.46 (d, J = 7.2 Hz, 3H), 7.26 (t, J = 4.0 Hz, 1H), 6.65 (dd, J = 8.2, 2.5 Hz, 1H), 6.44–6.31 (m, 2H), 3.82 (s, 3H), 2.25 (s, 3H). NMR data are in agreement with those from the literature.[3]

***N*-(4-Methoxyphenyl)-1-phenylethan-1-imine (3o)**



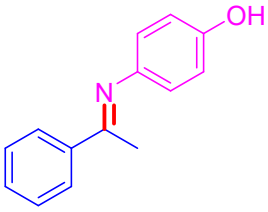
Yellow solid (105.9 mg, 94% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.00–7.93 (m, 2H), 7.48–7.41 (m, 3H), 6.95–6.88 (m, 2H), 6.79–6.73 (m, 2H), 3.82 (s, 3H), 2.25 (s, 3H). NMR data are in agreement with those from the literature.[3]

***N*-(3-Methoxyphenyl)-1-phenylethan-1-imine (3p)**



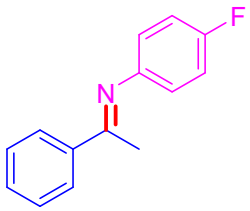
Yellow oil (101.4 mg, 90% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.01 (dd, J = 7.6, 2.1 Hz, 2H), 7.50–7.43 (m, 3H), 7.23–7.15 (m, 2H), 7.05–6.90 (m, 1H), 6.65 (d, J = 7.7 Hz, 1H), 2.17 (s, 3H), 2.10 (s, 3H). NMR data are in agreement with those from the literature.[4]

***N*-(4-Hydroxyphenyl)-1-phenylethan-1-imine (3q)**



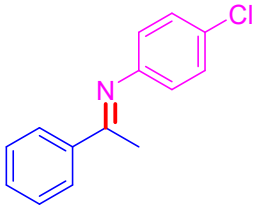
Yellow solid (94.0 mg, 89% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.97–7.90 (m, 2H), 7.46–7.42 (m, 3H), 6.84–6.78 (m, 2H), 6.72–6.67 (m, 2H), 2.62 (s, 1H), 2.25 (s, 3H). NMR data are in agreement with those from the literature.[5]

***N*-(4-Fluorophenyl)-1-phenylethan-1-imine (3r)**



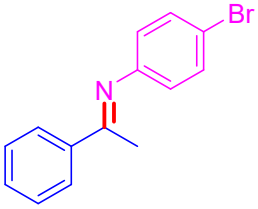
Yellow solid (87.4 mg, 82% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.00–7.91 (m, 2H), 7.52–7.40 (m, 3H), 7.05 (t, J = 8.7 Hz, 2H), 6.80–6.70 (m, 2H), 2.24 (s, 3H). NMR data are in agreement with those from the literature.[1]

***N*-(4-Chlorophenyl)-1-phenylethan-1-imine (3s)**



Yellow solid (97.6 mg, 85% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.98–7.94 (m, 2H), 7.49–7.43 (m, 3H), 7.32 (d, J = 8.6 Hz, 2H), 6.76–6.71 (m, 2H), 2.23 (s, 3H). NMR data are in agreement with those from the literature.[1]

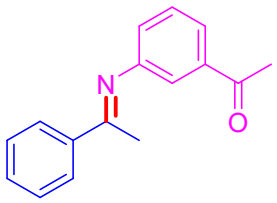
***N*-(4-Bromophenyl)-1-phenylethan-1-imine (3t)**



Yellow solid (111.0 mg, 81% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.00–7.91 (m, 2H), 7.50–7.42 (m, 5H), 6.72–6.65 (m, 2H), 2.23 (s, 3H). NMR data are in agreement

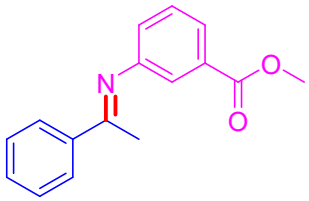
with those from the literature.[6]

***N*-(3-Acetylphenyl)-1-phenylethan-1-imine (3u)**



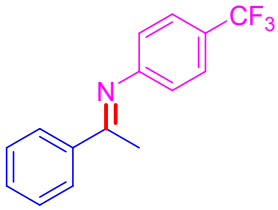
Yellow solid (99.7 mg, 84% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.02–7.93 (m, 2H), 7.80–7.65 (m, 1H), 7.50–7.42 (m, 4H), 7.40 (t, J = 1.9 Hz, 1H), 7.05–6.98 (m, 1H), 2.61 (s, 3H), 2.24 (s, 3H). NMR data are in agreement with those from the literature.[7]

***N*-[3-(Methoxycarbonyl)phenyl]-1-phenylethan-1-imine (3v)**



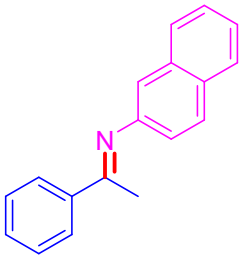
Yellow solid (103.9 mg, 82% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.01–7.93 (m, 2H), 7.99–7.95 (m, 1H), 7.56–7.37 (m, 5H), 7.03–6.98 (m, 1H), 3.92 (s, 3H), 2.23 (s, 3H). NMR data are in agreement with those from the literature.[7]

***N*-[4-(Trifluoromethyl)phenyl]-1-phenylethan-1-imine (3w)**



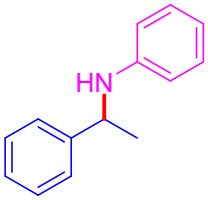
Yellow solid (105.3 mg, 80% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.02–7.93 (m, 2H), 7.60 (d, J = 8.2 Hz, 2H), 7.54–7.42 (m, 3H), 6.91–6.84 (m, 2H), 2.23 (s, 3H). NMR data are in agreement with those from the literature.[7]

***N*-(Naphthalen-2-yl)-1-phenylethan-1-imine (3x)**



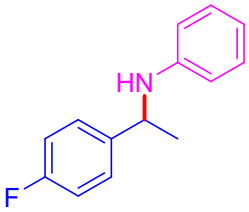
Yellow solid (106.7 mg, 87% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.03–7.98 (m, 2H), 7.83 (t, J = 8.5 Hz, 2H), 7.77 (d, J = 8.2 Hz, 1H), 7.50–7.42 (m, 4H), 7.05–7.36 (m, 1H), 7.16 (d, J = 2.1 Hz, 1H), 7.06–7.01 (m, 1H), 2.27 (s, 3H). NMR data are in agreement with those from the literature.[8]

***N*-(1-Phenylethyl)aniline (4a)**



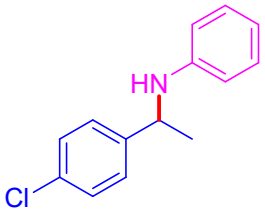
Colorless oil (88.8 mg, 90% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36 (d, J = 7.1 Hz, 2H), 7.31 (t, J = 7.6 Hz, 2H), 7.25–7.19 (m, 1H), 7.08 (dd, J = 8.6, 7.3 Hz, 2H), 6.64 (t, J = 7.3 Hz, 1H), 6.53–6.47 (m, 2H), 4.47 (q, J = 6.7 Hz, 1H), 4.03 (s, 1H), 1.50 (d, J = 6.8 Hz, 3H). NMR data are in agreement with those from the literature.[9]

***N*-(1-(4-Fluorophenyl)ethyl)aniline (4g)**



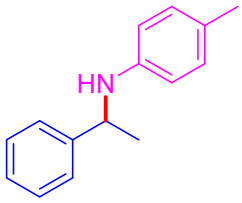
Yellow oil (90.4 mg, 84% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.34–7.30 (m, 2H), 7.11–7.06 (m, 2H), 7.02–6.96 (m, 2H), 6.68–6.63 (m, 1H), 6.50–6.47 (m, 2H), 4.46 (d, J = 6.7 Hz, 1H), 4.02 (s, 1H), 1.49 (d, J = 6.7 Hz, 3H). NMR data are in agreement with those from the literature.[10]

***N*-(1-(4-Chlorophenyl)ethyl)aniline (4h)**



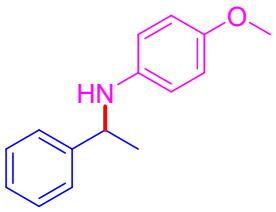
Colorless oil (93.8 mg, 81% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.33–7.25 (m, 4H), 7.09 (t, J = 7.8 Hz, 2H), 6.66 (t, J = 7.3 Hz, 1H), 6.47 (d, J = 8.0 Hz, 2H), 4.45 (q, J = 6.7 Hz, 1H), 4.04 (s, 1H), 1.49 (d, J = 6.7 Hz, 3H). NMR data are in agreement with those from the literature.[9]

4-Methyl-*N*-(1-phenylethyl)aniline (4l)



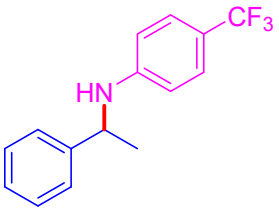
Yellow oil (90.9 mg, 86% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.37–7.33 (m, 2H), 7.32–7.27 (m, 2H), 7.23–7.17 (m, 1H), 6.89 (d, J = 8.3 Hz, 2H), 6.42 (d, J = 8.4 Hz, 2H), 4.44 (q, J = 6.7 Hz, 1H), 2.18 (s, 3H), 1.49 (d, J = 6.7 Hz, 3H). NMR data are in agreement with those from the literature.[9]

4-Methoxy-*N*-(1-phenylethyl)aniline (4o)



Yellow oil (100.0 mg, 88% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.37–7.34 (m, 2H), 7.33–7.28 (m, 2H), 7.25–7.18 (m, 1H), 6.68 (d, J = 8.7 Hz, 2H), 6.47 (d, J = 8.8 Hz, 2H), 4.40 (q, J = 6.6 Hz, 1H), 3.68 (s, 3H), 1.49 (d, J = 6.9 Hz, 3H). NMR data are in agreement with those from the literature.[9]

***N*-(1-Phenylethyl)-[4-(trifluoromethyl)phenyl]aniline (4w)**



Colorless oil (99.5 mg, 75% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.37–7.28 (m, 6H), 7.27–7.21 (m, 1H), 6.49 (d, J = 8.5 Hz, 2H), 4.50 (q, J = 6.7 Hz, 1H), 4.36 (s, 1H), 1.52 (d, J = 6.7 Hz, 3H). NMR data are in agreement with those from the literature.[11]

6. References

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Aldimines to Secondary Amines Using Iron-Catalysed Hydrosilylation. *Org. Biomol. Chem.* **2018**, *16*, 9368–9372.

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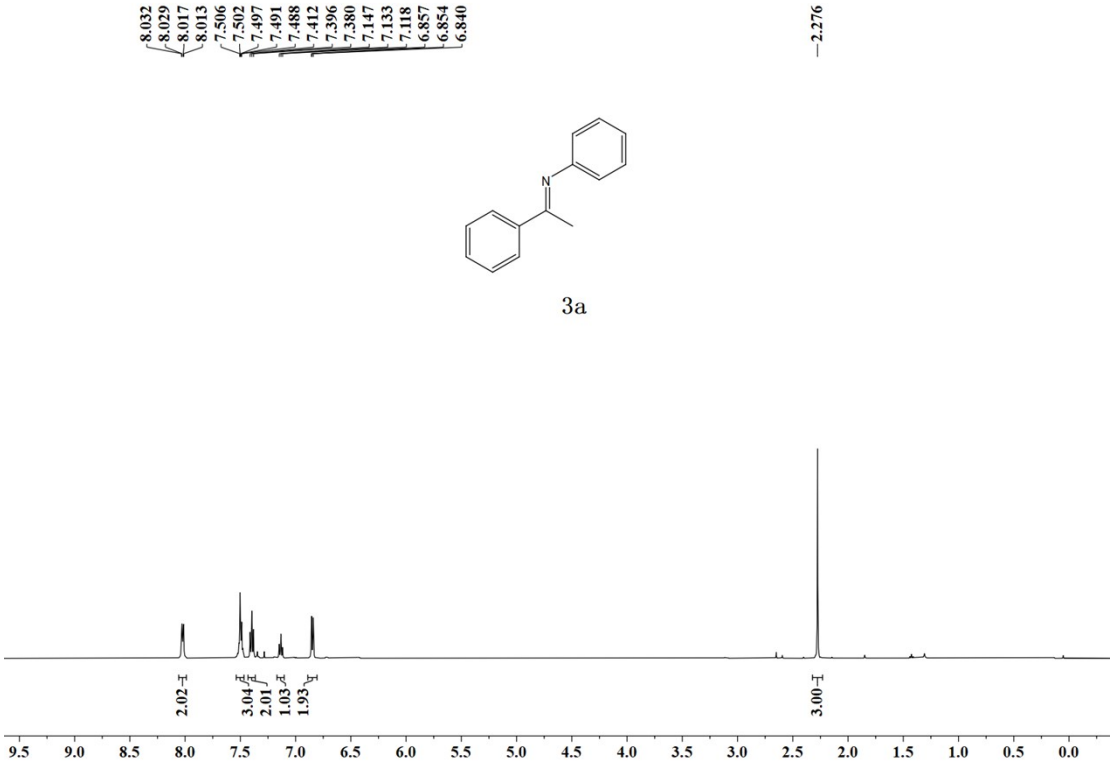
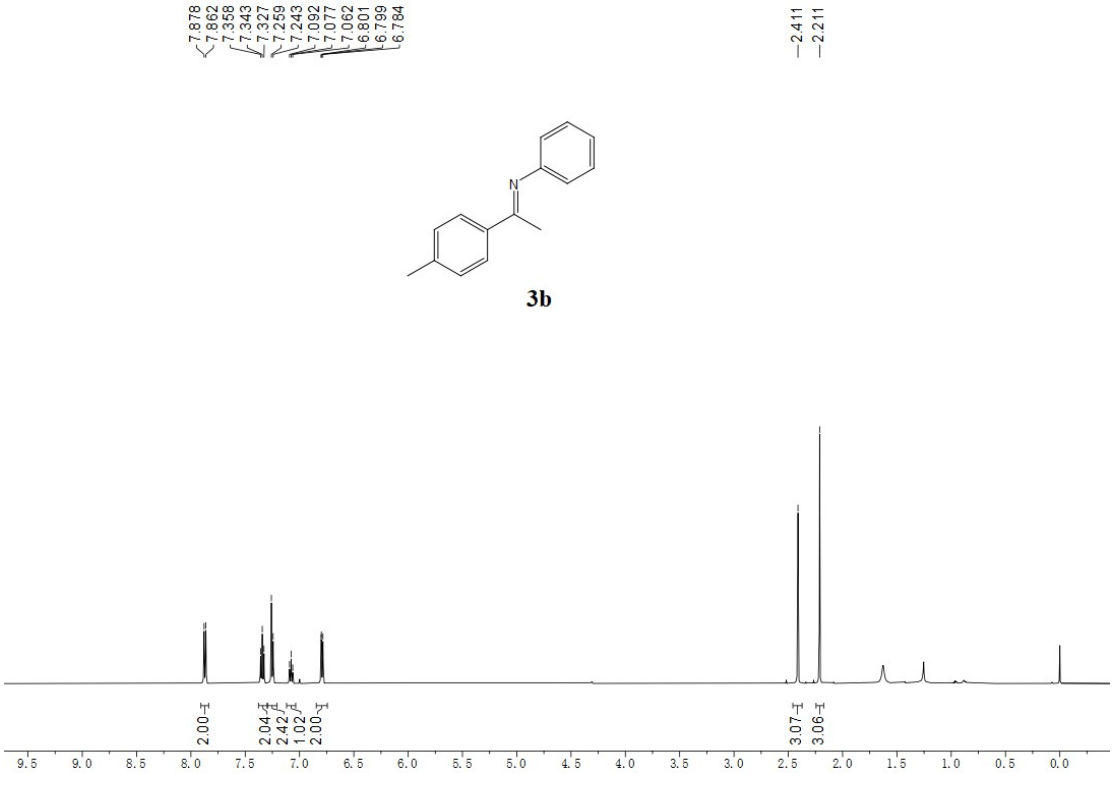
7. Copies of ^{1}H Spectra of Products

^{1}H NMR, 500 MHz, $CDCl_3$



3a

^{1}H NMR, 500 MHz, $CDCl_3$



3b

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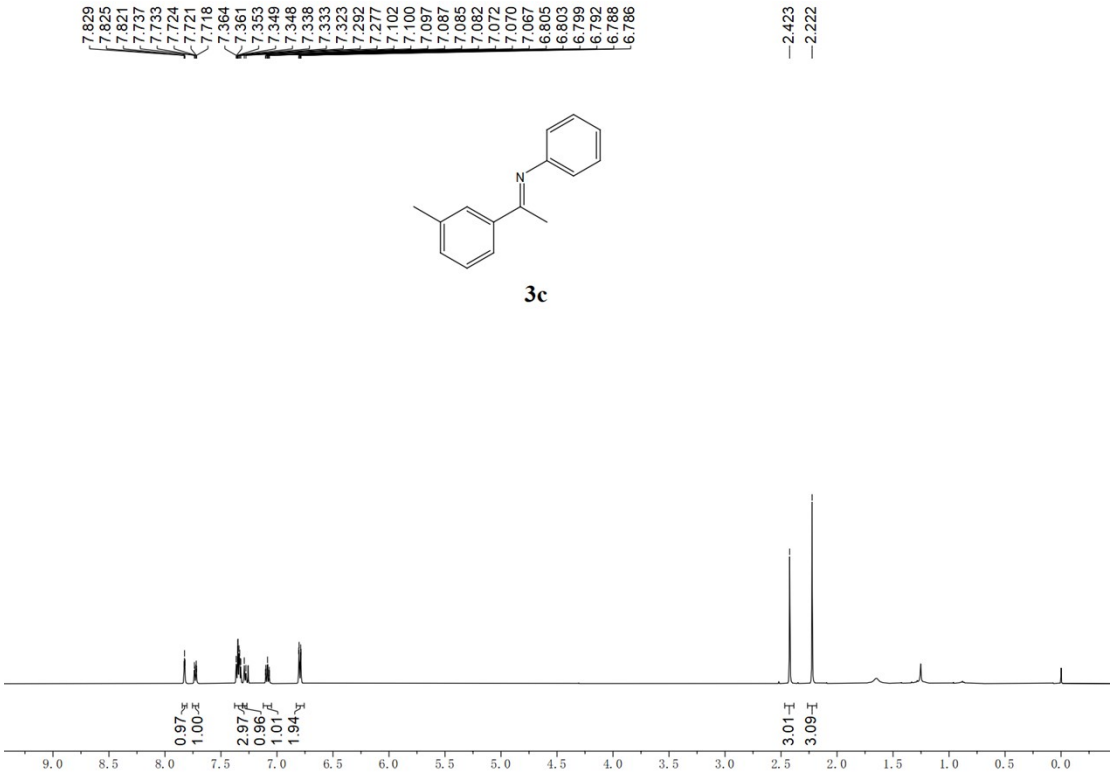

3c

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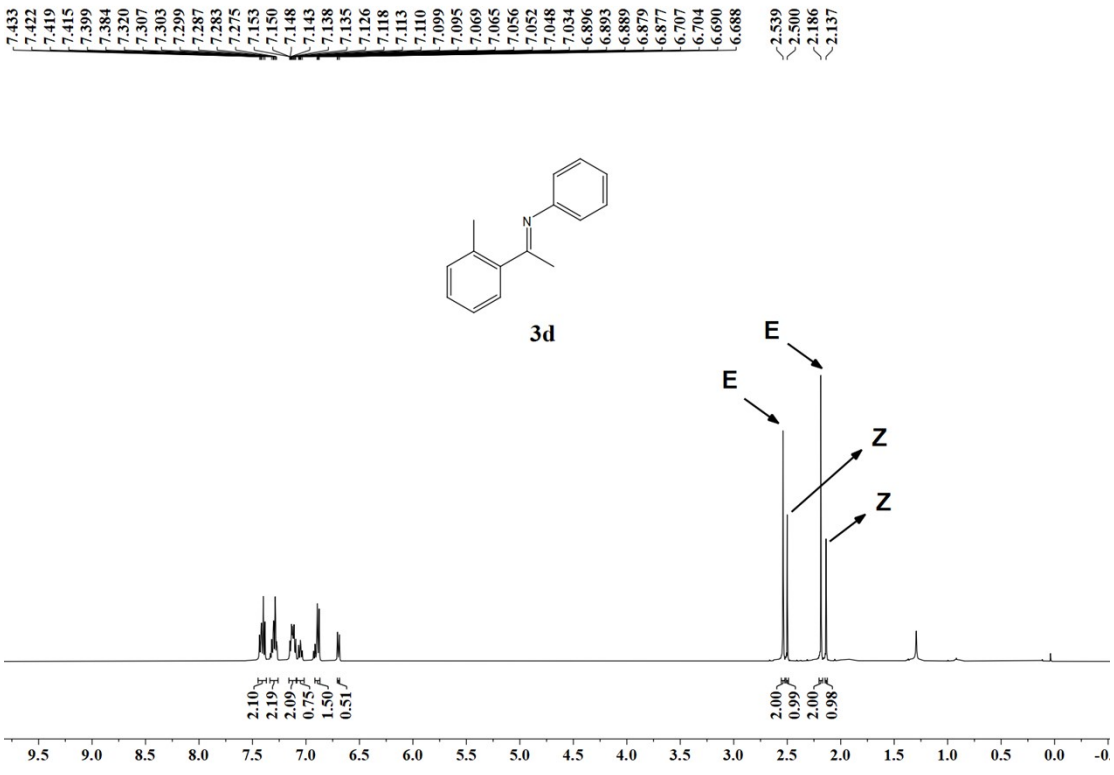

3d

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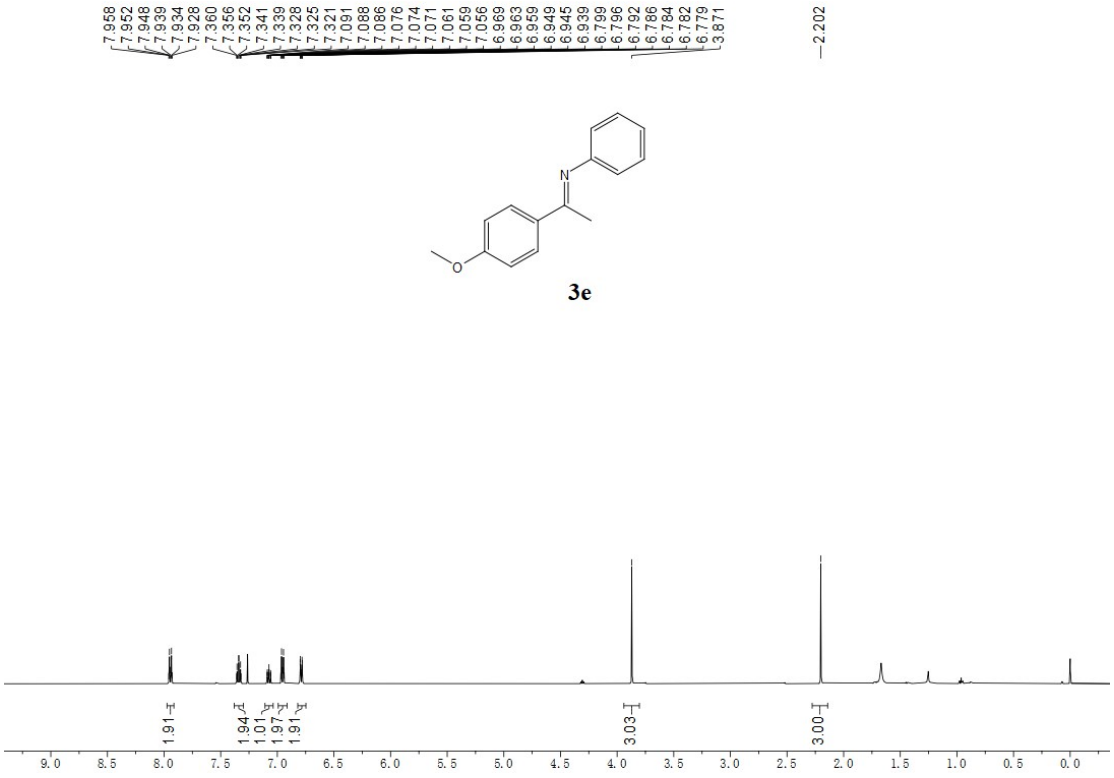

3e

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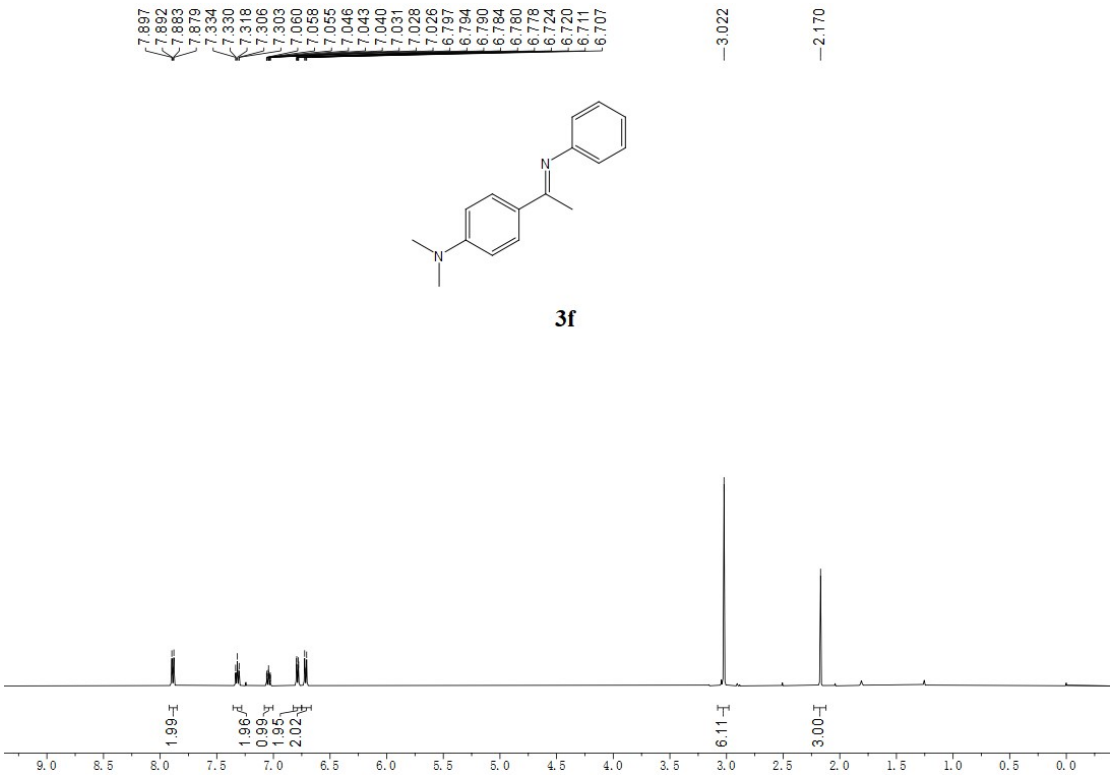

3f

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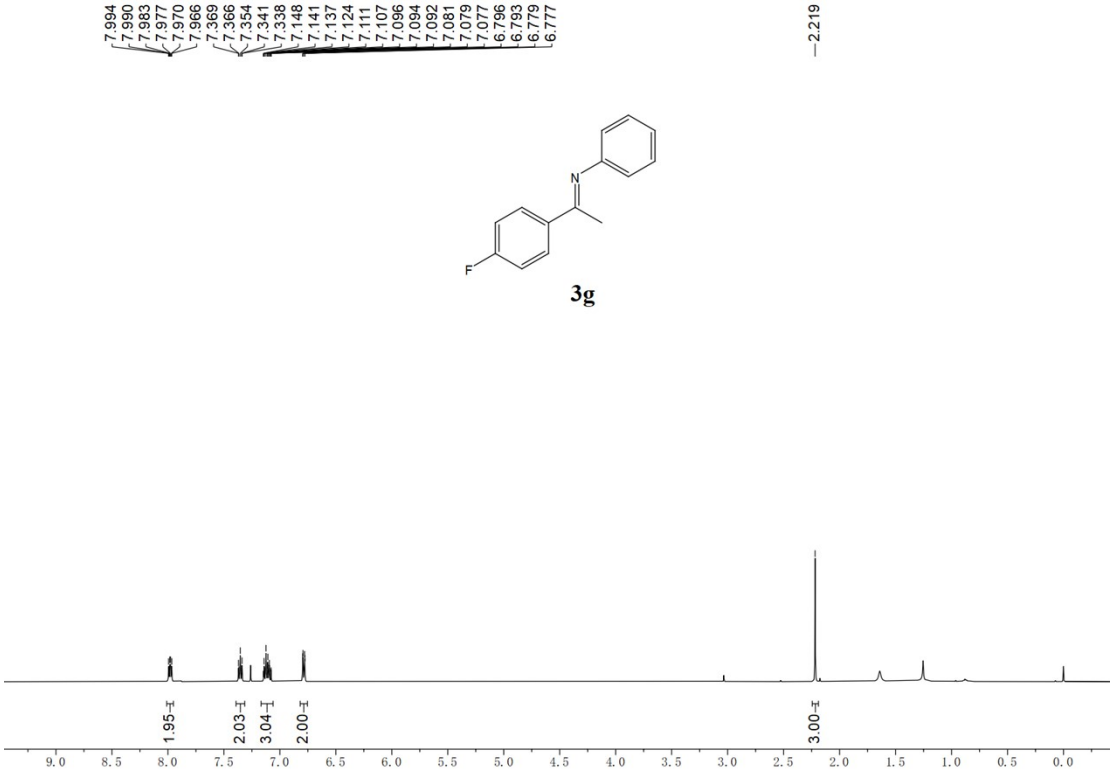

3g

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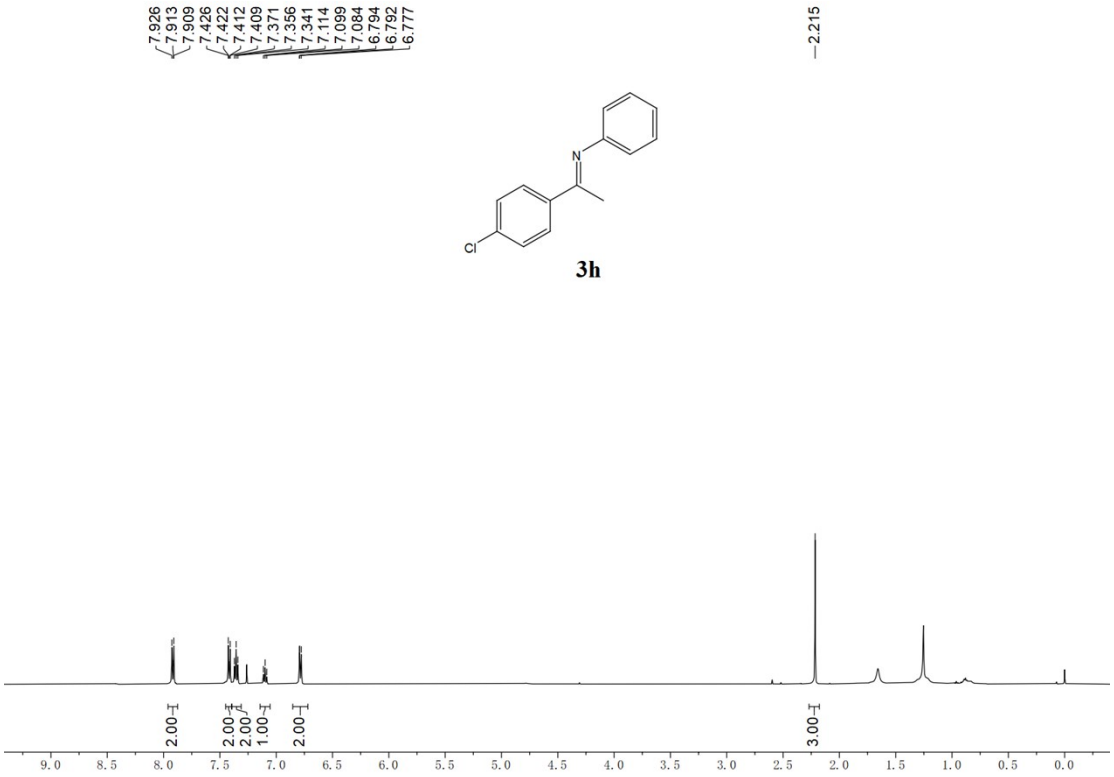

3h

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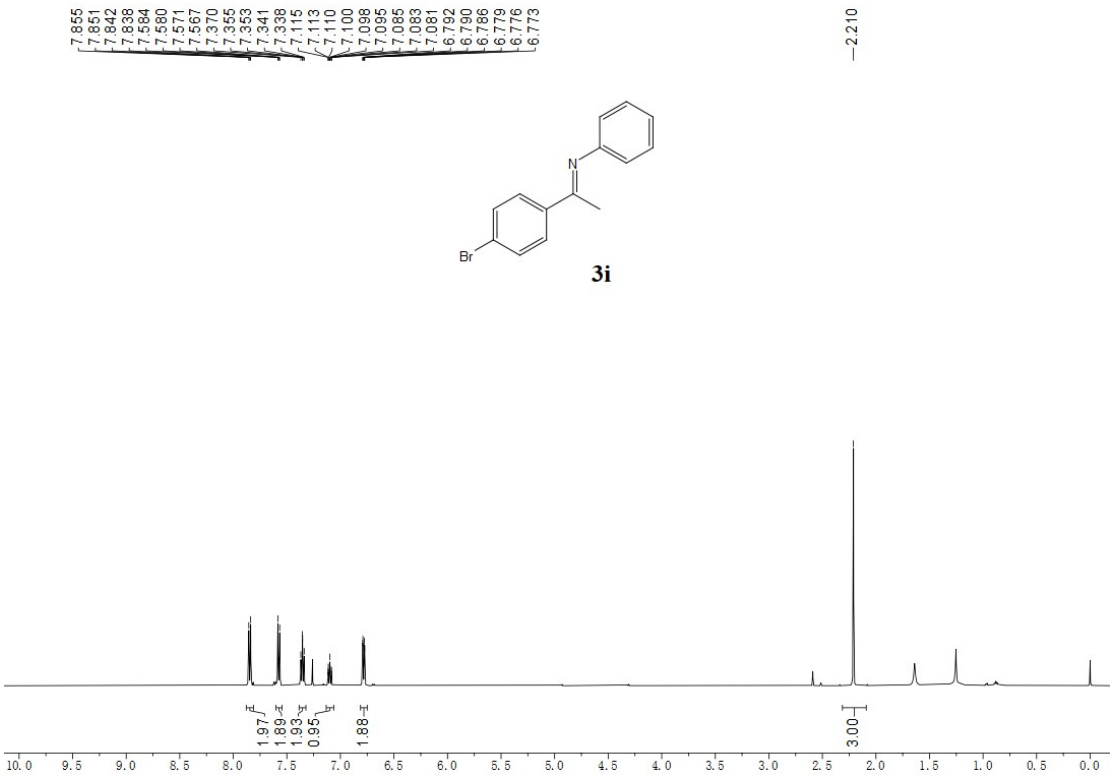

3i

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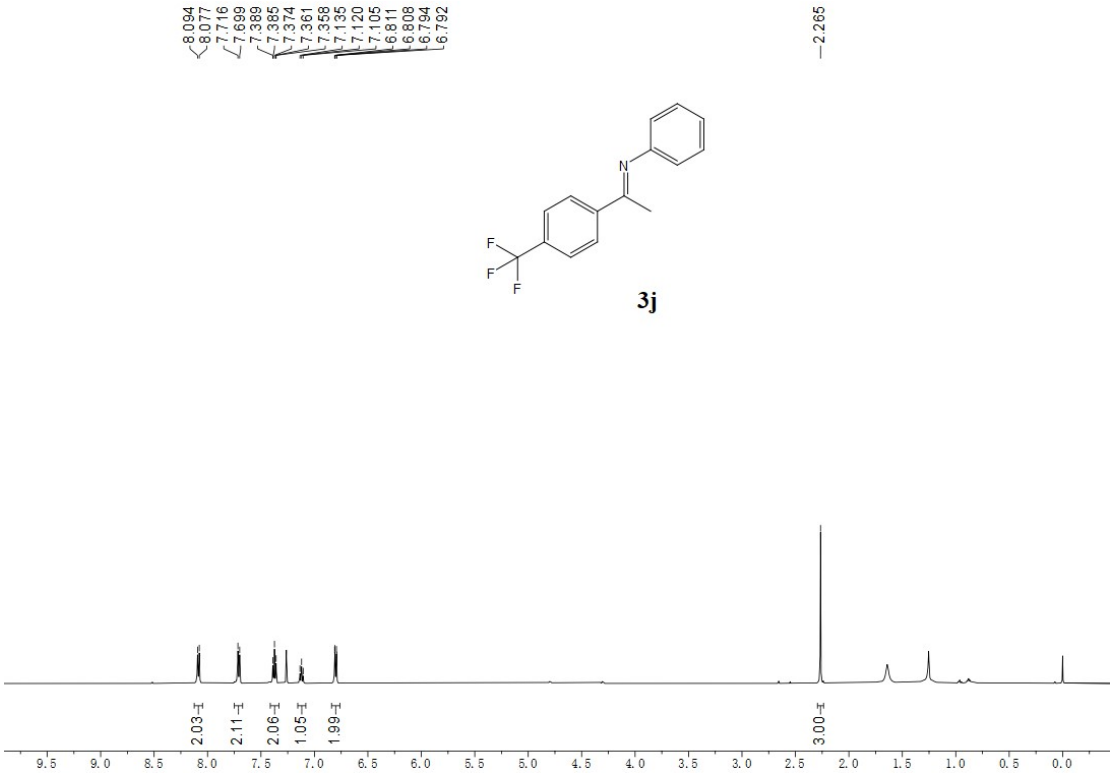

3j

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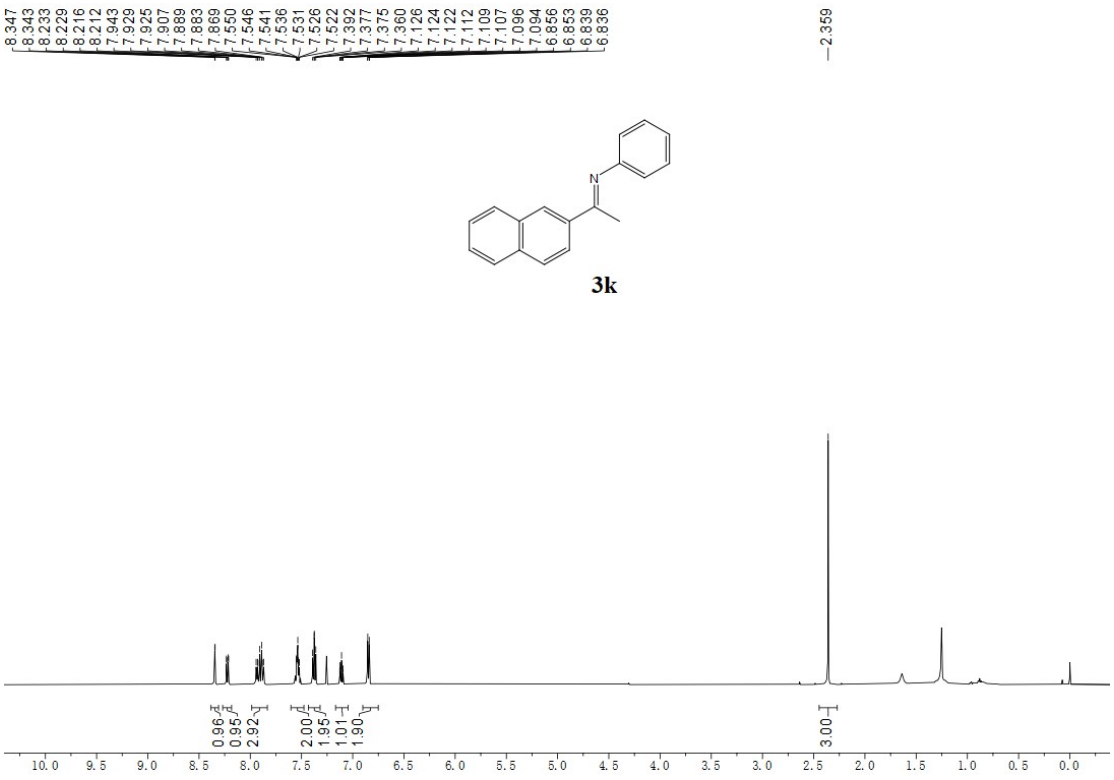

3k

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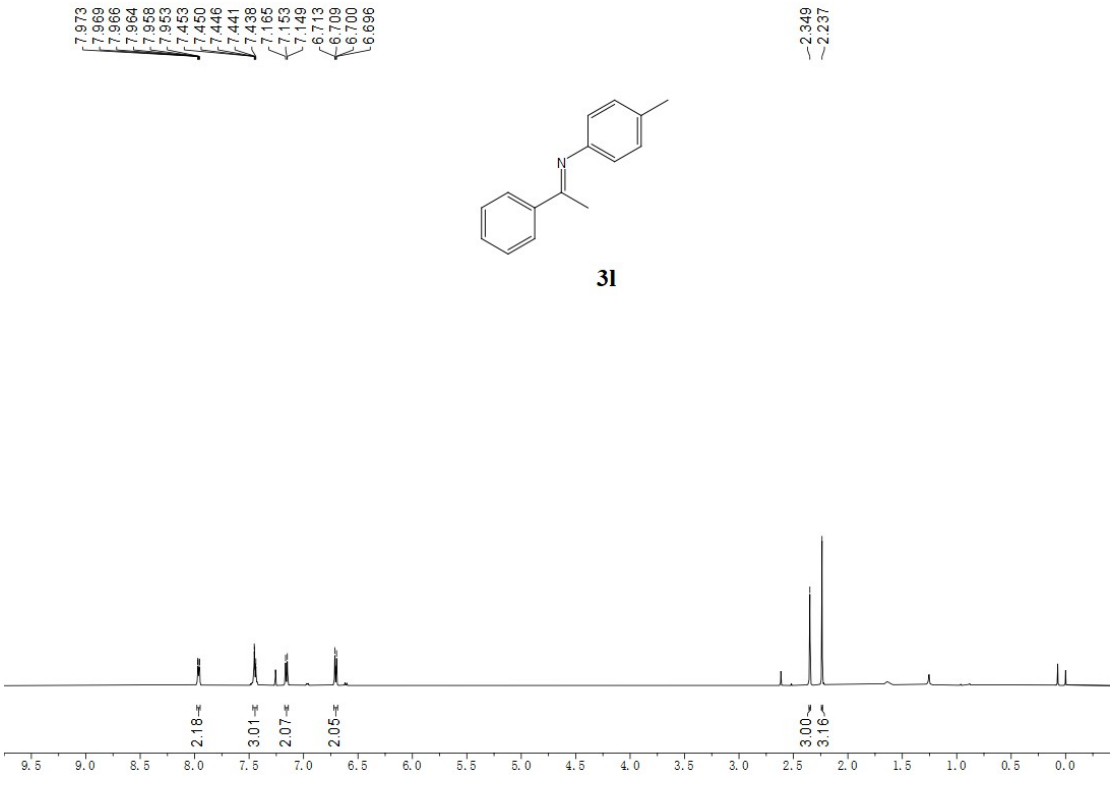

3l

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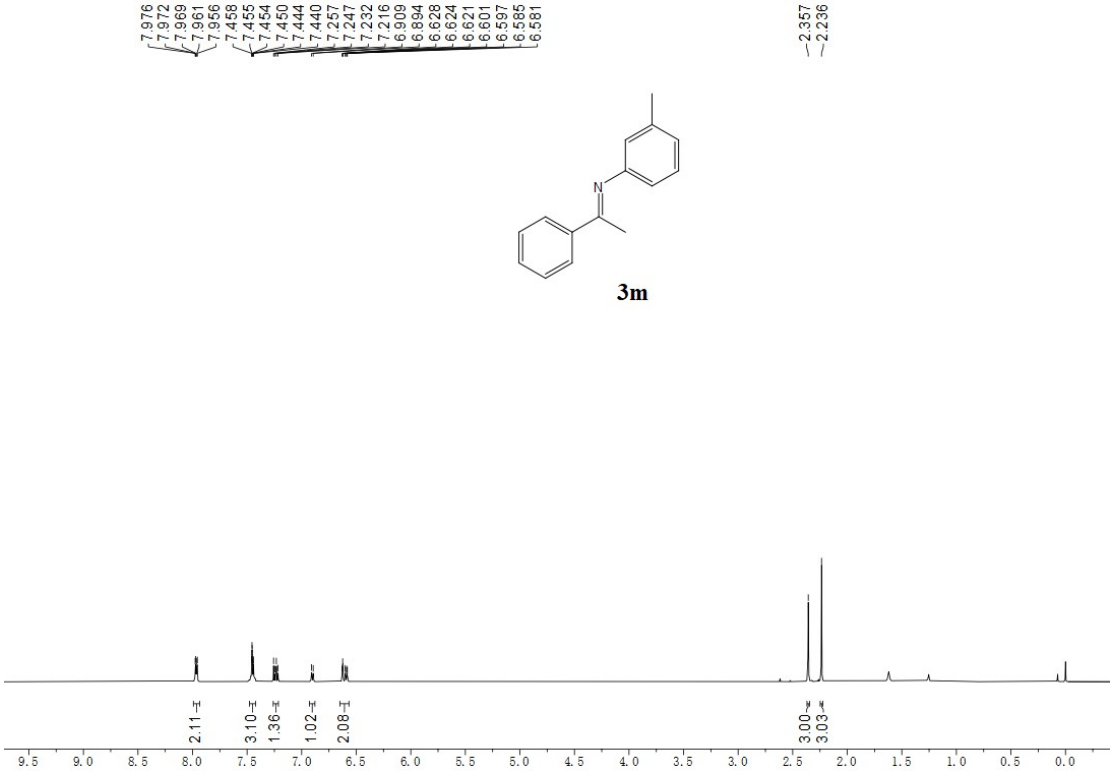

3m

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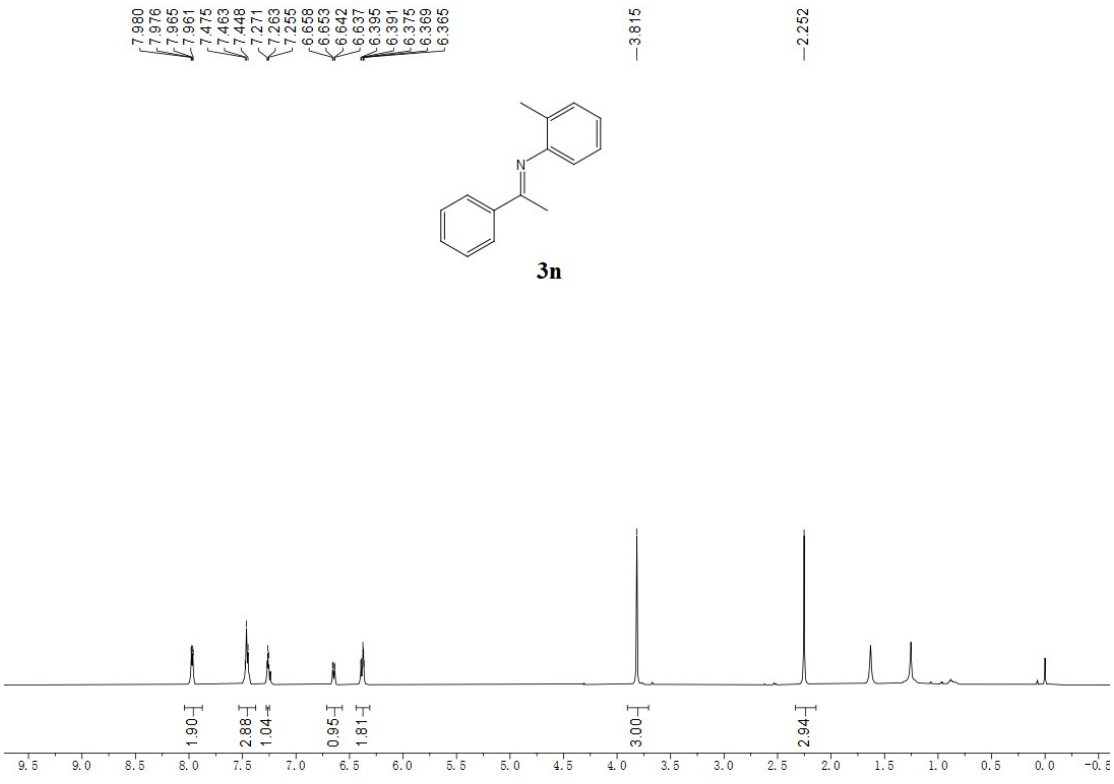

3n

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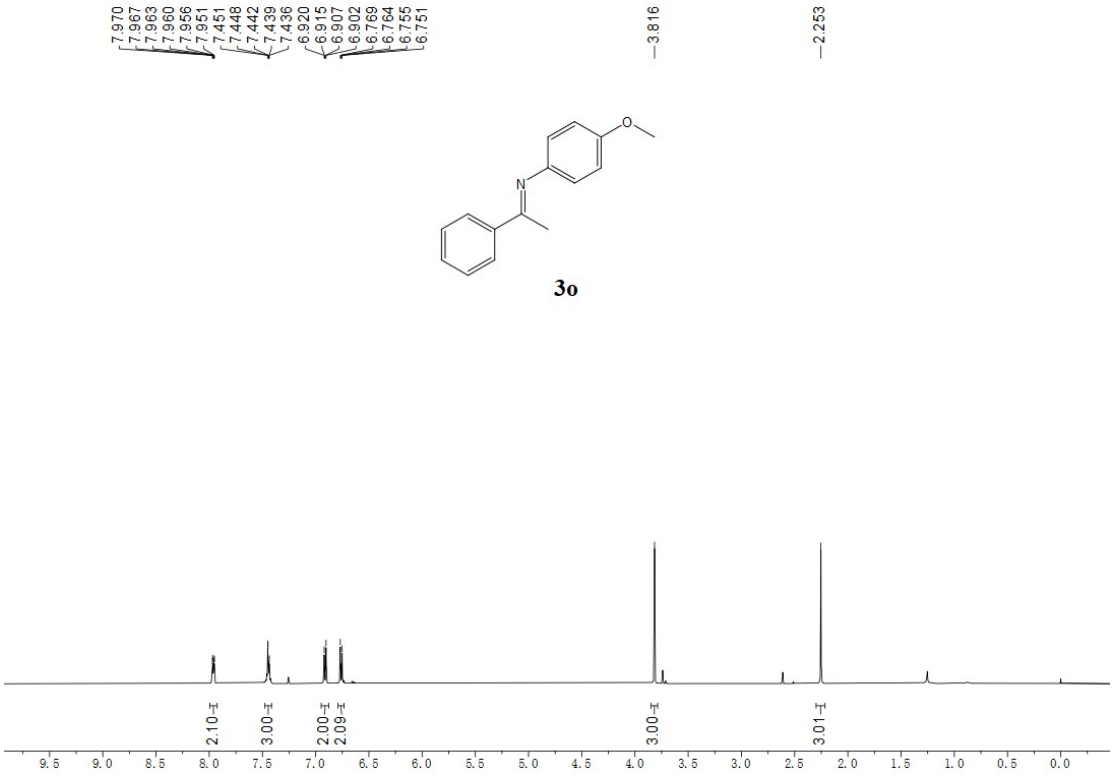

3o

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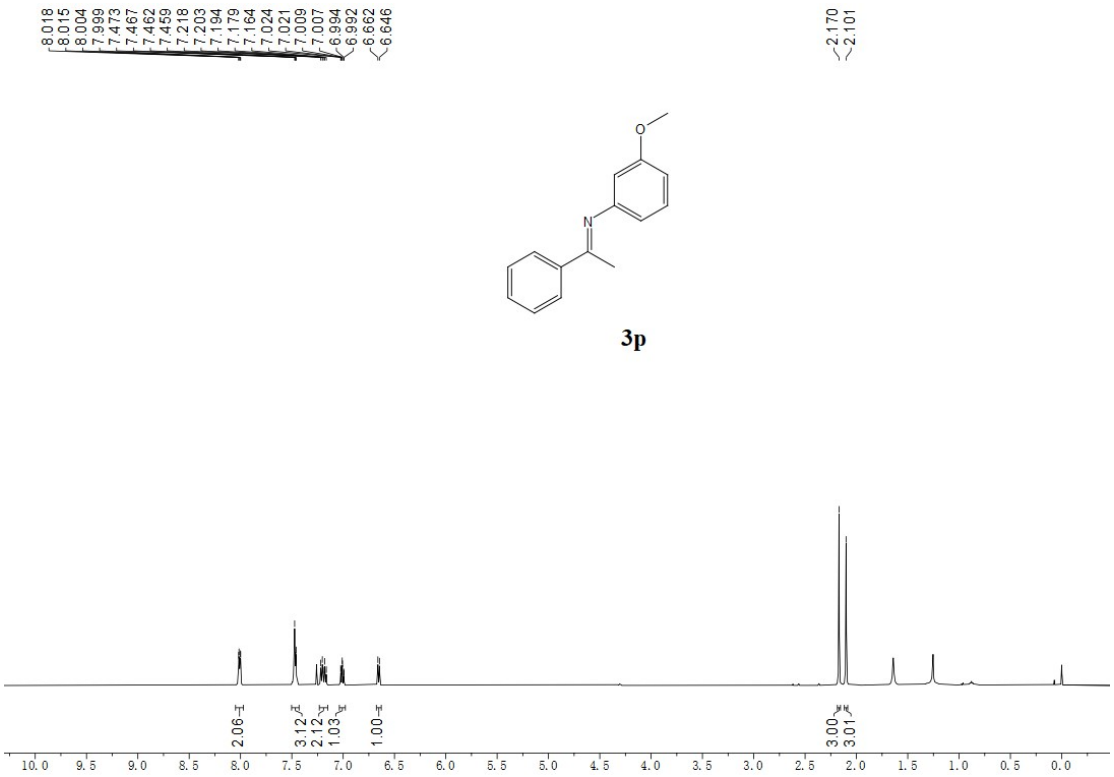

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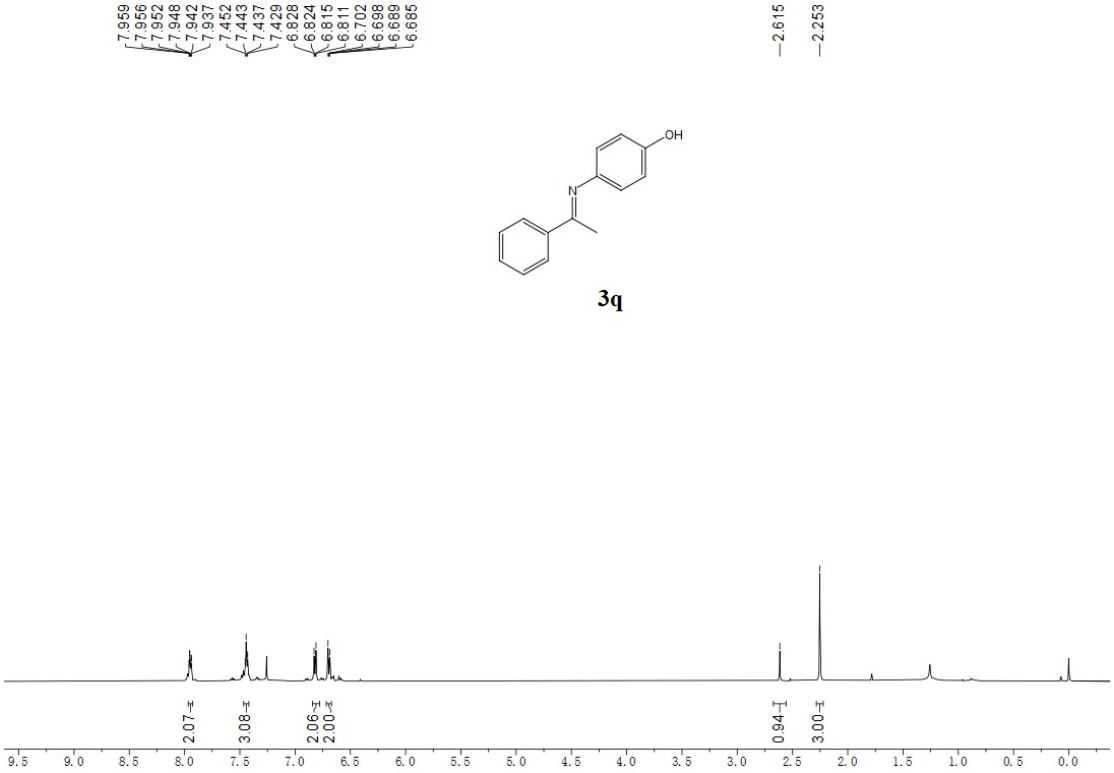

3q

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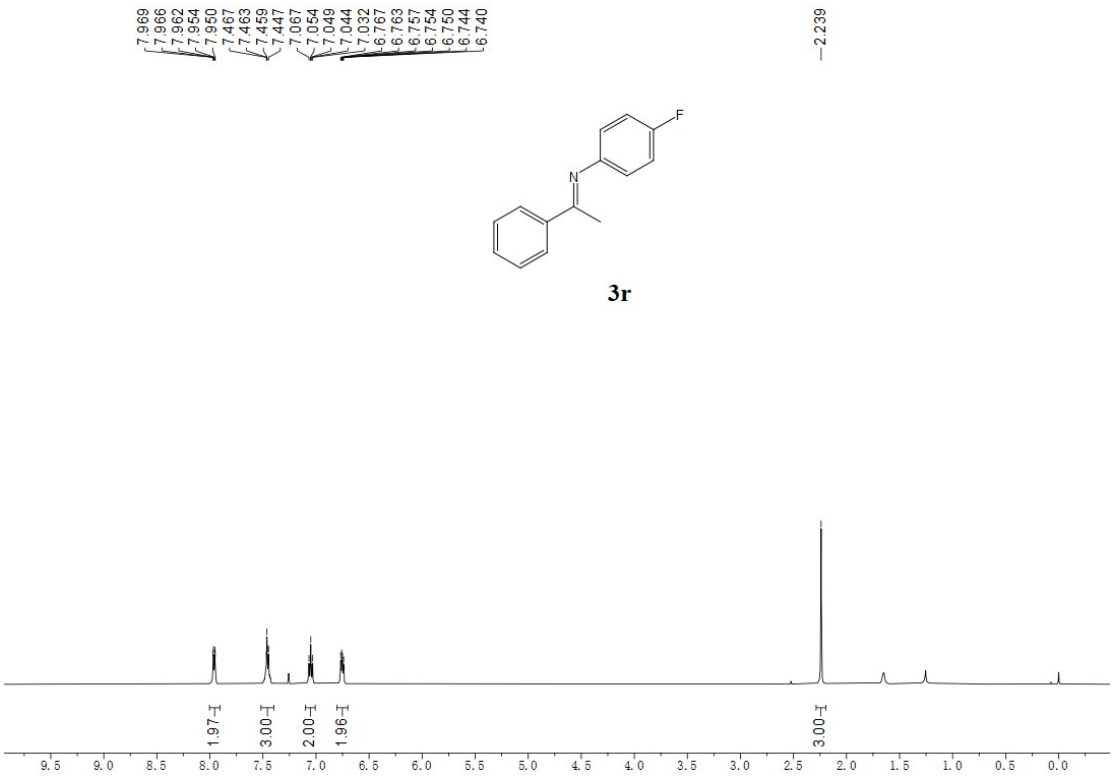

3r

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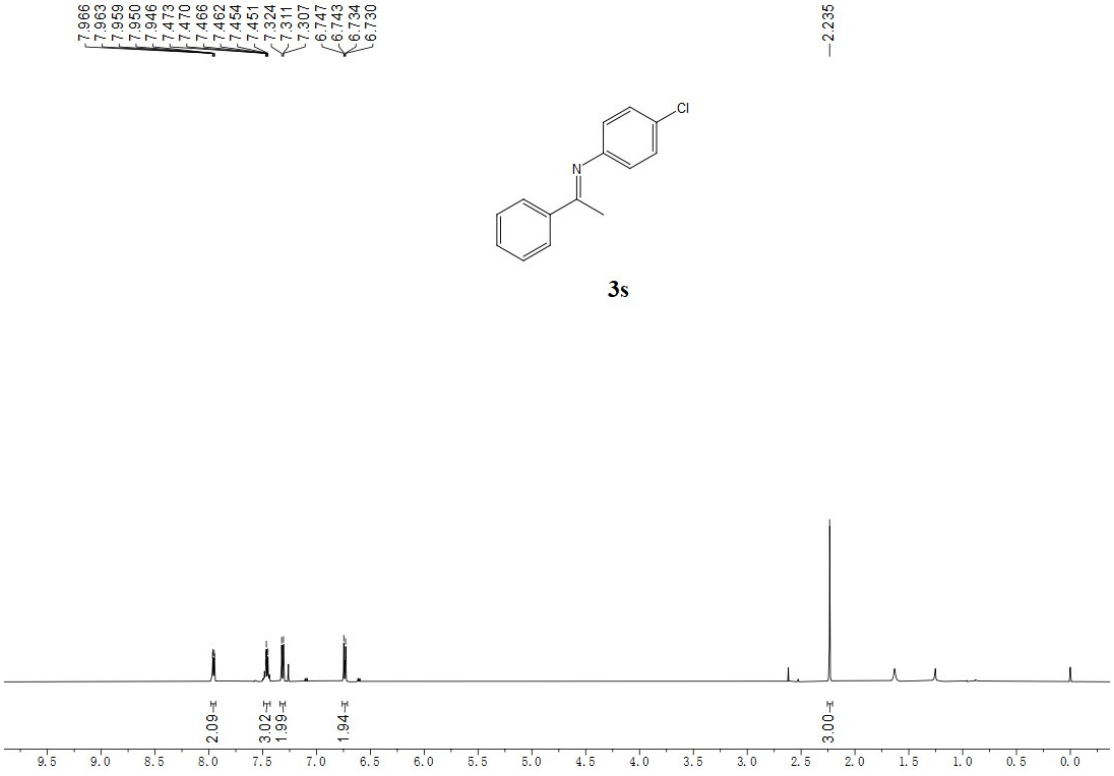

3s

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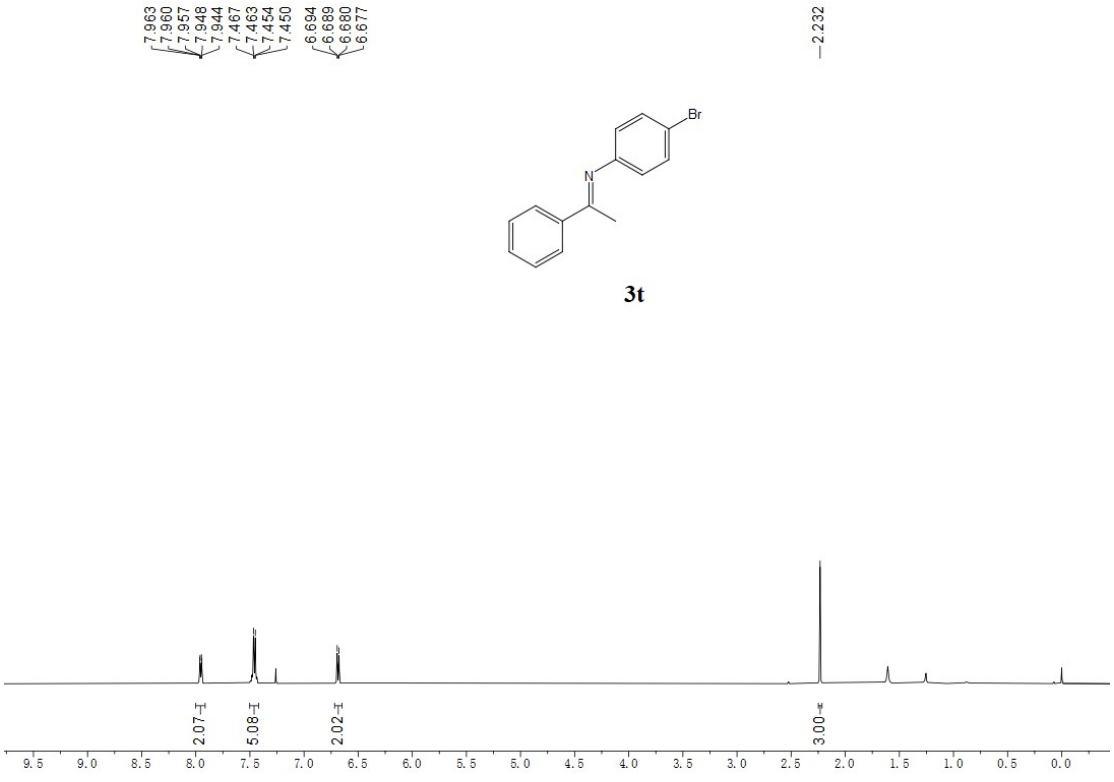

3t

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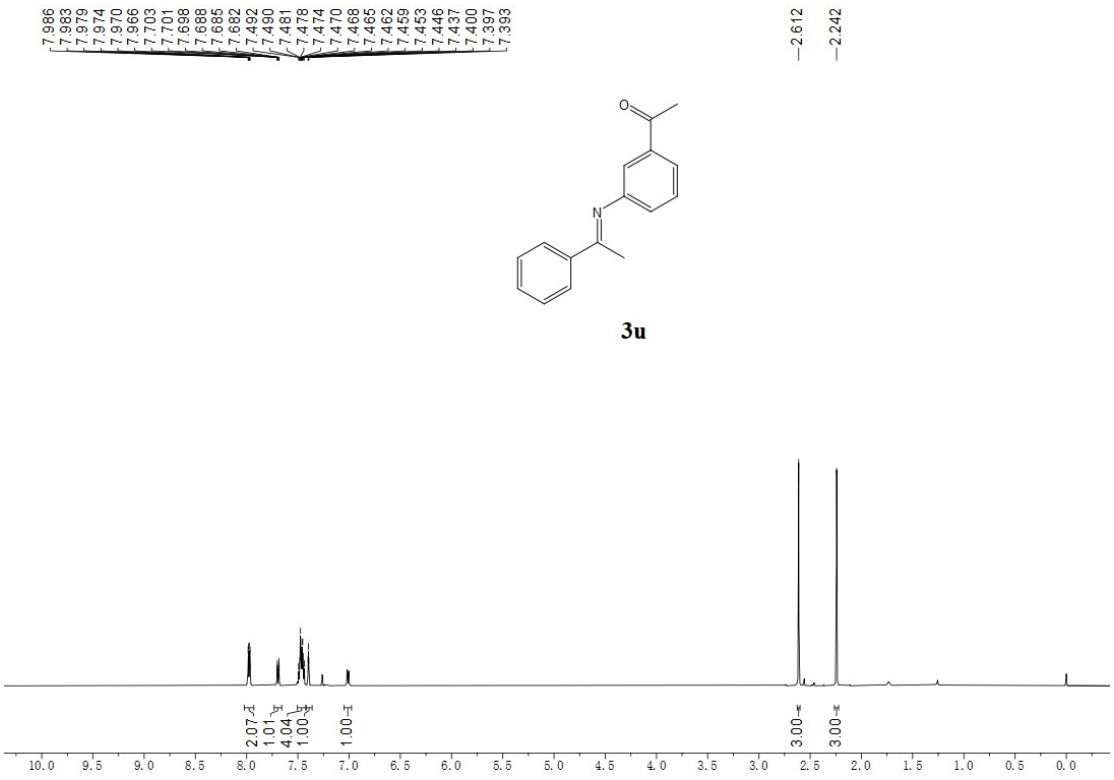

3u

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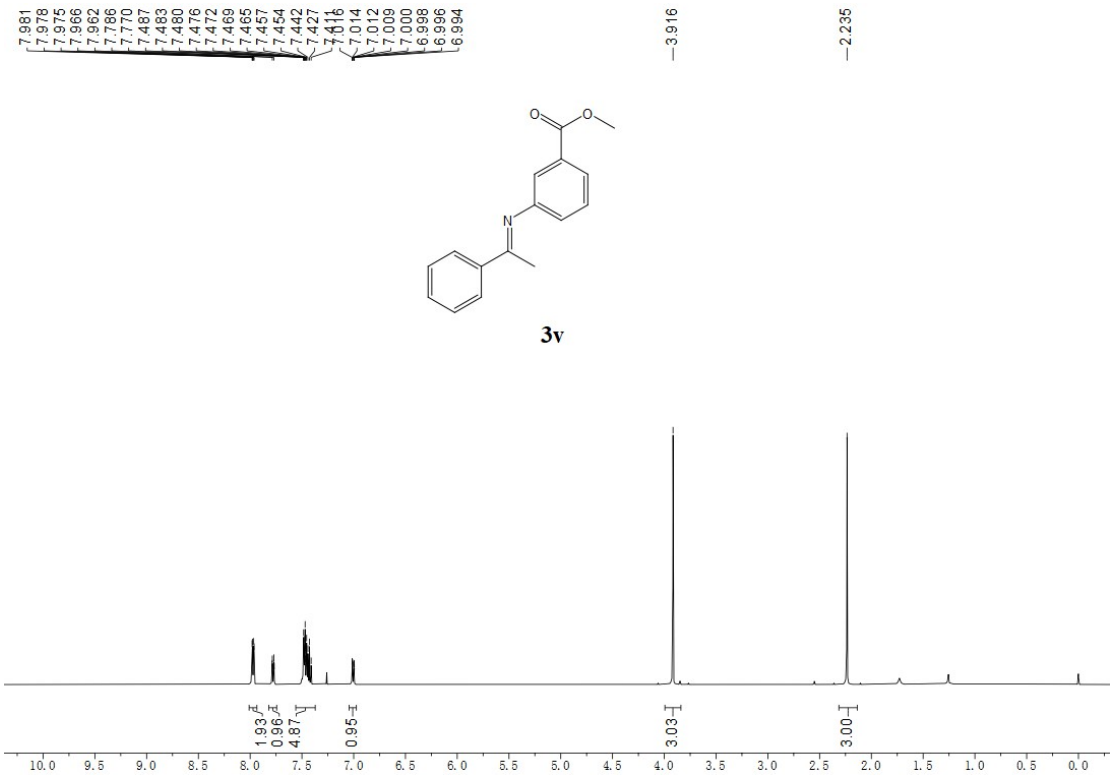

3v

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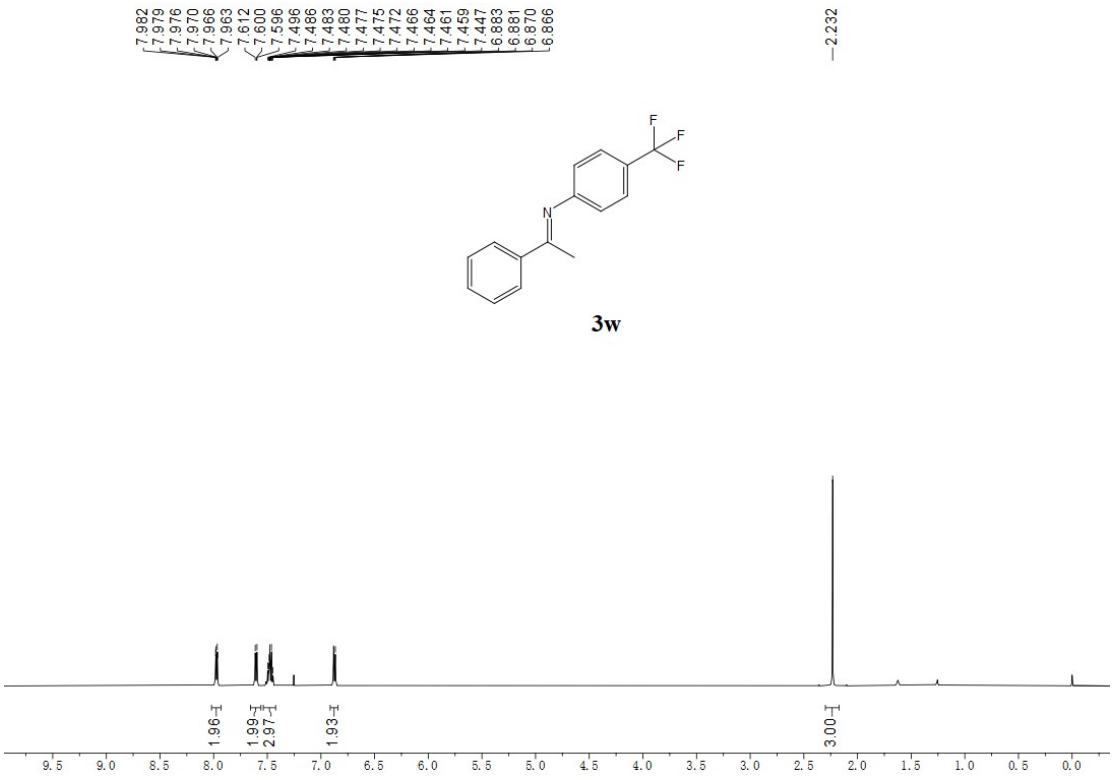

3w

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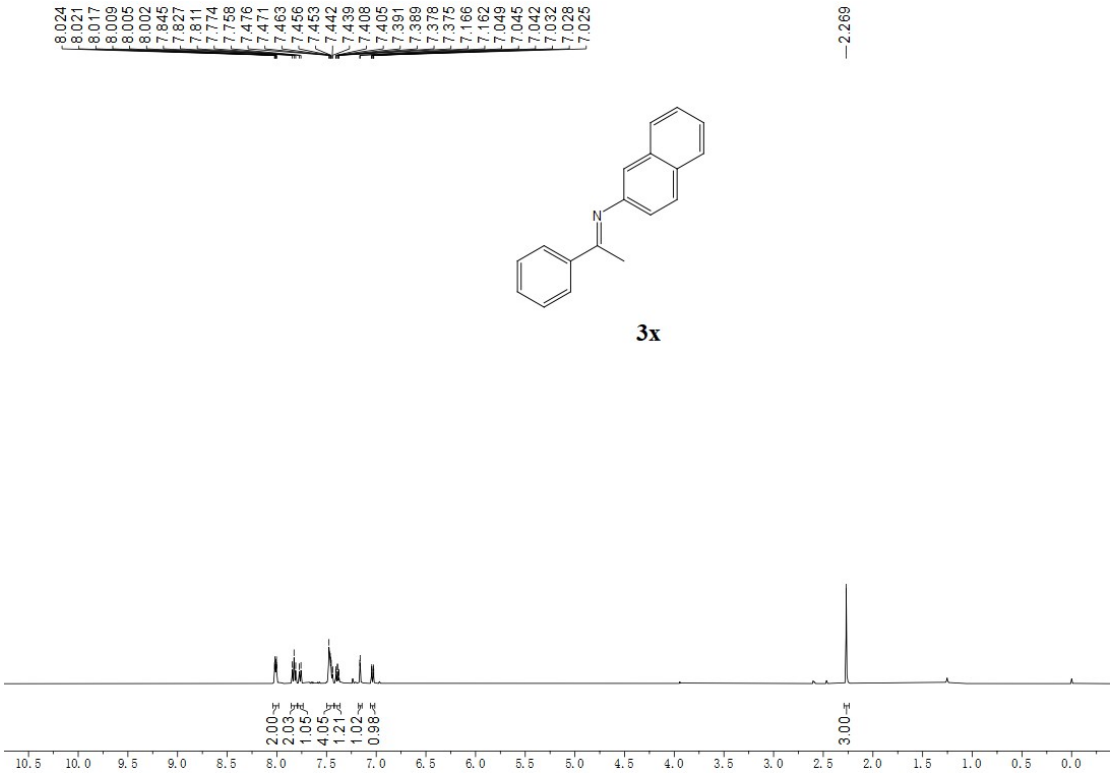

3x

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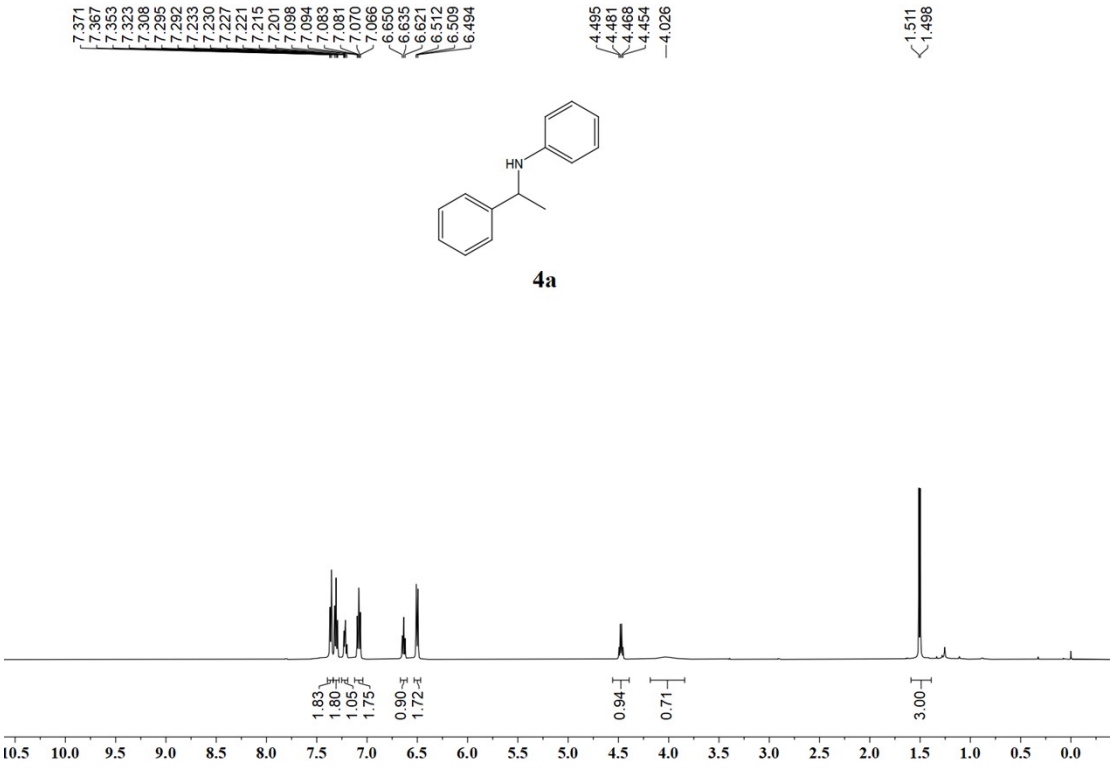

4a

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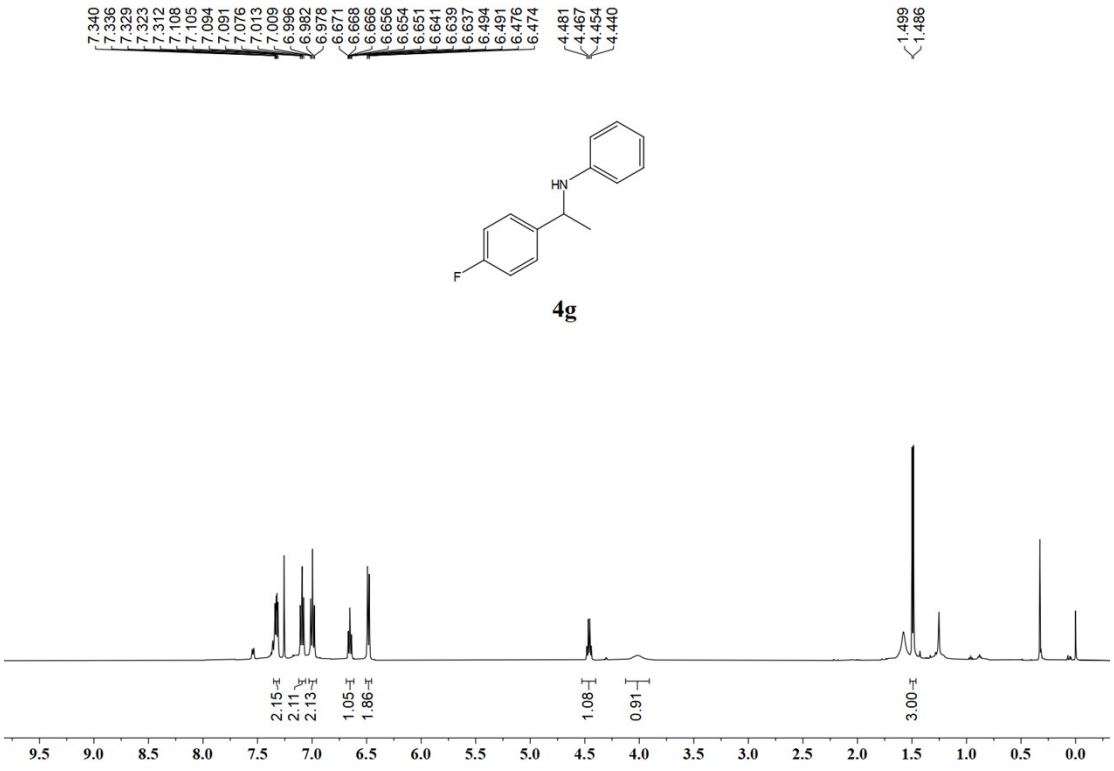

4g

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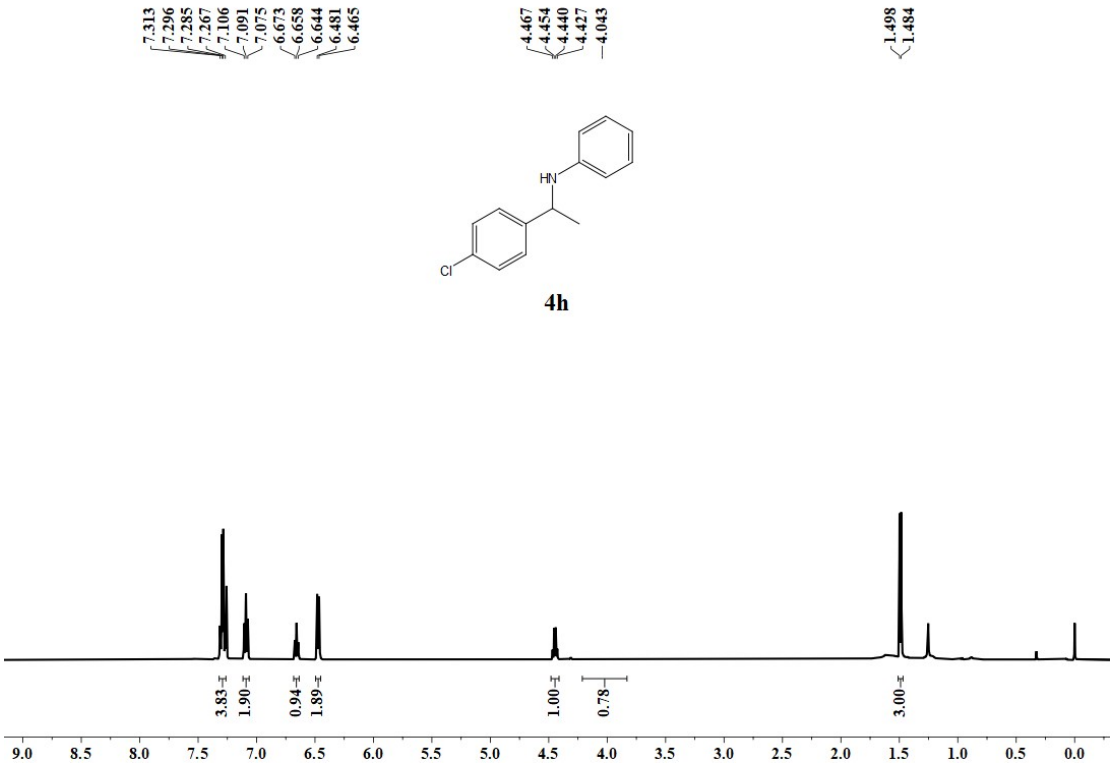

4h

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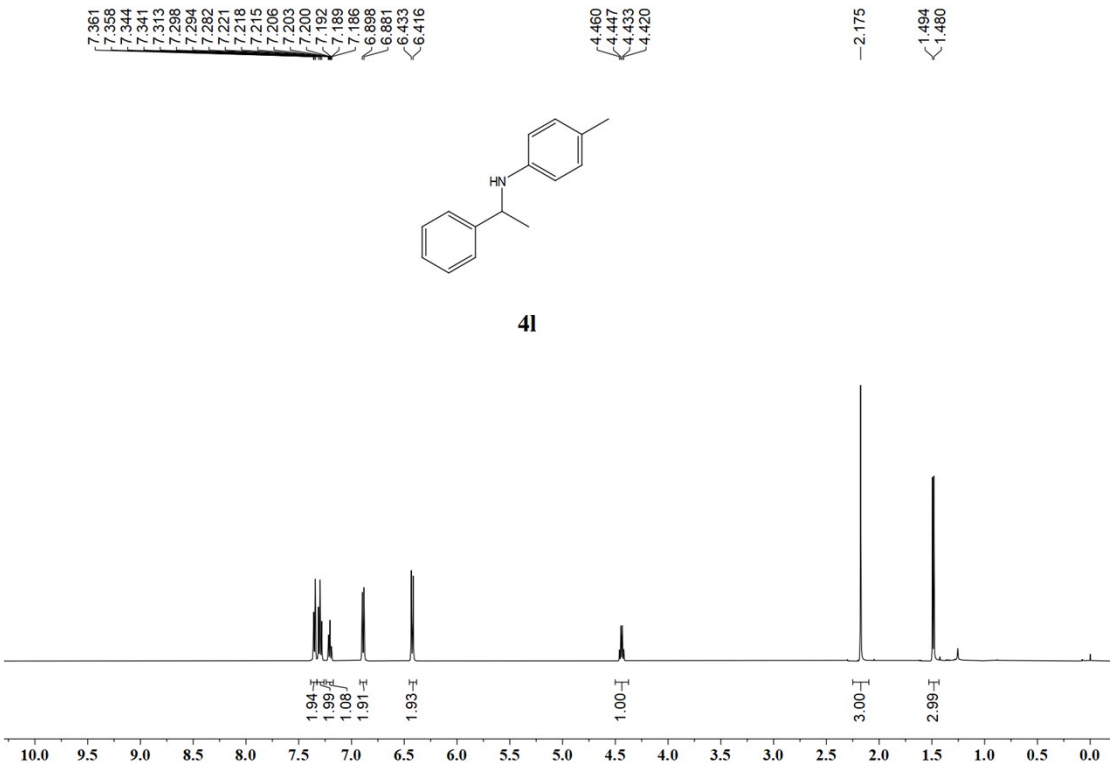

4l

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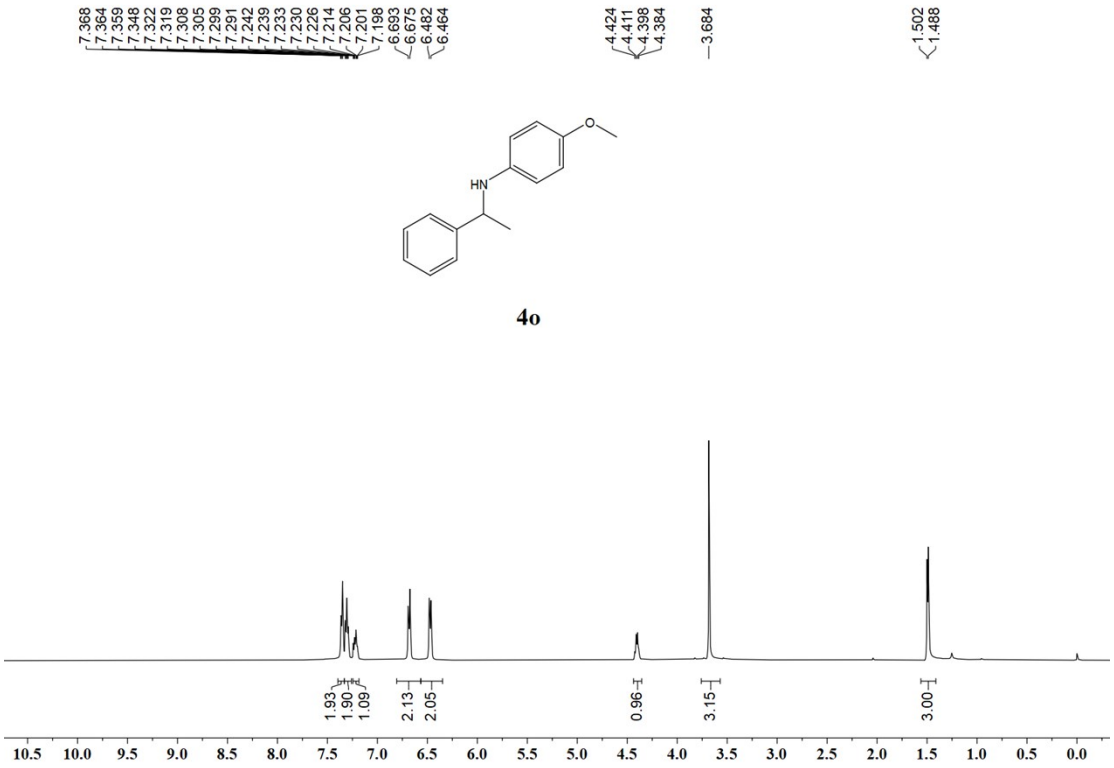

4o

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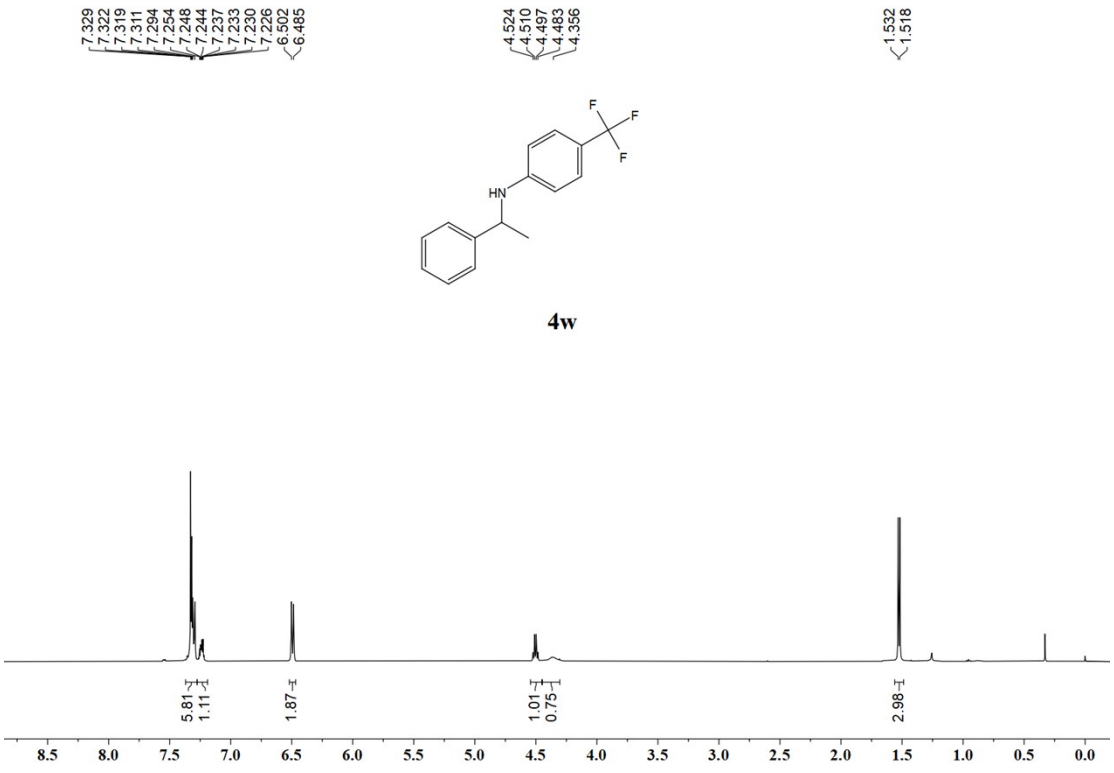

4w