Supporting Information

Selective amination of aryl chlorides catalyzed by Ni(PMe₃)₄

Shumiao Zhang, Xiaoyan Li, Hongjian Sun*

School of Chemistry and Chemical Engineering, Key Laboratory of Special Functional Aggregated Materials,

Ministry of Education, Shandong University, Shanda Nanlu 27, 250199 Jinan, PR China

General information

All the reactions for C-N cross-coupling were carried out using standard Schlenk techniques under N₂ atmosphere. Toluene was dried by Na using benzophenone as indicator and distilled under N₂. CH₃CN, DMSO and DMF were refluxed with CaH₂ and distilled under N₂. Other chemicals obtained commercially were used as received without further purification. ¹H NMR, ¹³C NMR and ¹⁹F NMR were recorded on 300 or 400 MHz Bruker instruments. Chemical shifts were referenced by external standard expressing in ppm (tetramethysilane, 0 ppm, CDCl₃).

General procedure for synthesis of the chloro-substituted Schiff bases (1a-1i)

The imines were prepared from 1:1 ratio of corresponding aldehyde and amine in ethanol. **1a-1f** were recrystallized in pentane. **1g-1i** are gained as yellow oil. The scope of the substrates is shown in Table S1.



General procedure for C-N coupling reactions (Procedure A)

To a sealed tube was added 1 (1.5 mmol), amines, NaOH and ${}^{n}Bu_{4}NBr$ (0.075 mmol). Then the tube was evacuated and recharged by N₂ for three times. Toluene (4 mL) and Ni(PMe₃)₄ (0.060 mmol in its toluene solution)was added under N₂ atmosphere by syringe. The sealed tube was put in a 80°C oil bath. After reaction completed, the solvent was removed in vacuo completely and the product was extracted by pentane (15mL*3). Pure products were obtained by recrystallation from pentane, producing yellow powder or crystals.

General procedure for reducing of imines (Procedure B)

After the reaction of C-N coupling finished, solvent was removed and the crude products were extracted by pentane (15mL*3). After pentane was removed in vacuo, the imines were dissolved in 10mL of petroleum ether. Few drops of acetic acid was

added. Then $NaBH_4$ was added in a 30 mg amount over an interval of 2 minutes until the yellow color of imines faded into colorless. The precipitate was filtered off, and the solvent was concentrated. The products were obtained by silica gel column chromatography (petroleum ether : ethyl acetate 10:1).

Analytical data for 2a-2q

N-(4-methylphenyl)-2-[[(4-methylphenyl)imino]methyl]-3-chlorobenzenamine (2a).



Following procedure A, **1a** (0.396 g, 1.5 mmol) was used as substrate. The amount of 4-methylaniline and NaOH are both 1.5 mmol. After 12h heating, product **2a** (0.477 g, 95%) is obtained.

¹H NMR (300 MHz, CDCl₃) δ 11.76 (s, 1H), 9.26 (s, 1H), 7.25-7.15 (m, 8H), 7.12-7.05 (m, 2H), 6.76 (dd, ${}^{3}J_{\text{HH}} = 5.7\text{Hz}$, ${}^{4}J_{\text{HH}} = 2.7\text{Hz}$, 1H), 2.38 (s, 3H), 2.35 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 159.88, 149.53, 149.17, 138.89, 138.53, 136.74, 134.47, 132.70, 130.65, 130.55, 124.22, 121.81, 118.48, 114.82, 112.55, 21.68, 21.58. HRMS-ESI (m/z) Calcd for C₂₁H₁₉ClN₂ [M+H]: 335.1315; Found: 335.1260.



N-(2-chlorophenyl)-2-[[(2-chlorophenyl)imino]methyl]-3chloro-benzenamine (**2b**).

Following procedure A, **1b** (0.427 g, 1.5 mmol) was used as substrate. The amount of 2-chloroaniline and NaOH are both 1.5 mmol. After 12h heating, product **2b** (0.490 g, 87%) was

obtained.

¹H NMR (300 MHz, CDCl₃) δ 11.77 (s, 1H), 9.24 (s, 1H), 7.51-7.44 (m, 3H), 7.35-7.23 (m, 2H), 7.21-7.13 (m, 3H), 7.08 (td, ${}^{3}J_{\text{HH}} = 7.8$, ${}^{4}J_{\text{HH}} = 1.5$ Hz, 1H), 7.02 (d, ${}^{3}J_{\text{HH}} = 8.4$ Hz, 1H), 6.85 (dd, ${}^{3}J_{\text{HH}} = 7.8$, ${}^{4}J_{\text{HH}} = 0.9$ Hz, 1H). ¹³C NMR (75 MHz, CDCl₃) δ 160.67, 148.10, 148.07, 138.46, 137.88, 132.63, 130.52, 130.04, 129.23, 129.00, 127.80, 127.26, 127.07, 125.24, 124.77, 119.94, 118.94, 115.19, 112.78. HRMS-ESI (m/z) Calcd for C₁₉H₁₃Cl₃N₂ [M+H]: 375.0223; Found: 375.0136.

N-phenyl-2-[(phenylimino)methyl]-3-chloro-benzenamine (2c).



Following procedure A, **1c** (0.375 g, 1.5 mmol) was used as substrate. The amount of aniline and NaOH are both 1.5 mmol. After 12h heating, product **2c** (0.405 g, 88%) was obtained.

¹H NMR (300 MHz, CDCl₃) δ 11.82 (s, 1H), 9.26 (s, 1H), 7.45-7.28 (m, 8H), 7.25-7.19(m, 2H), 7.15-7.10(m, 2H), 6.81 (dd, ${}^{3}J_{\text{HH}} = 7.5$, ${}^{4}J_{\text{HH}} = 1.2$ Hz, 1H). ¹³C NMR (75 MHz, CDCl₃) δ 160.00, 150.94, 148.36, 140.51, 138.38, 132.26, 129.42, 129.29, 126.24, 123.96, 123.05, 121.28, 118.27, 114.37, 112.10. HRMS-ESI (m/z) Calcd for C₁₉H₁₅ClN₂ [M+H]: 307.1002; Found: 307.0953.



N-(4-chlorophenyl)-2-[[(4-

chlorophenyl)imino]methyl]-3-chloro-benzenamine (2d).

Following procedure A, 1d (0.427 g, 1.5 mmol) was

used as substrate. The amount of 4-chloroaniline and NaOH are both 1.5 mmol. After 12h heating, product **2d** (0.513 g, 91%) was obtained.

¹H NMR (300 MHz, CDCl₃) δ 11.66 (s, 1H), 9.22 (s, 1H), 7.40-7.31 (m, 4H), 7.24-7.11 (m, 6H), 6.83 (dd, ${}^{3}J_{\text{HH}} = 6.9$, ${}^{4}J_{\text{HH}} = 2.1$ Hz, 1H). ¹³C NMR (75 MHz, CDCl₃) δ 160.53, 149.54, 148.41, 139.28, 138.81, 132.91, 132.27, 129.82, 129.72, 129.40, 124.53, 122.82, 119.08, 114.71, 112.37. HRMS-ESI (m/z) Calcd for C₁₉H₁₃Cl₃N₂ [M+H]: 375.0223; Found: 375.0133.

N-(2-chlorophenyl)-2-[[(4-methylphenyl)imino]methyl]-3-chloro-benzenamine (2e).



Following procedure A, **1a** (0.396 g, 1.5 mmol) was used as substrate. The amount of 2-chloroaniline and NaOH were both 1.5 mmol. After 12h heating, product **2e** (0.426 g, 80%) was obtained.

¹H NMR (300 MHz, CDCl₃) δ 12.13 (s, 1H), 9.28 (s, 1H), 7.55 (dd, ³*J*_{HH} = 8.1, ⁴*J*_{HH} =

1.5 Hz, 1H), 7.46 (dd, ${}^{3}J_{\text{HH}} = 7.8$, ${}^{4}J_{\text{HH}} = 1.2$ Hz, 1H), 7.24-7.12 (m, 7H), 7.00 (td, ${}^{3}J_{\text{HH}} = 7.8$, ${}^{4}J_{\text{HH}} = 1.2$ Hz, 1H), 6.87 (dd, ${}^{3}J_{\text{HH}} = 7.5$, ${}^{4}J_{\text{HH}} = 1.2$ Hz, 1H), 2.38 (s, 3H). 13 C NMR (100 MHz, CDCl₃) δ 158.27, 147.89, 146.77, 138.25, 138.17, 136.48, 131.85, 130.35, 129.94, 127.13, 126.97, 123.81, 121.96, 121.29, 119.39, 116.06, 112.68, 21.09. HRMS-ESI (m/z) Calcd for C₂₀H₁₆Cl₂N₂ [M+H]: 355.0769; Found: 355.0767.

N-phenyl-2-[[(4-methylphenyl)imino]methyl]-3-chloro-benzenamine (2f).



Following procedure A, **1a** (0.396 g, 1.5 mmol) was used as substrate. The amount of aniline and NaOH were both 1.5 mmol. After 12h heating, product **2f** (0.418g, 87%) was obtained.

¹H NMR (300 MHz, CDCl₃) δ 11.88 (s, 1H), 9.26 (s, 1H), 7.40-7.28 (m, 4H), 7.24-7.09 (m, 7H), 6.80 (dd, ${}^{3}J_{\text{HH}} = 7.5$, ${}^{4}J_{\text{HH}} = 1.2\text{Hz}$, 1H), 2.38 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 159.18, 148.39, 148.28, 140.66, 138.29, 136.23, 132.08, 129.94, 129.45, 123.89, 122.99, 121.17, 118.31, 114.54, 112.10, 21.07. HRMS-ESI (m/z) Calcd for C₂₀H₁₇ClN₂ [M+H]: 321.1159; Found: 321.1164.

N-(4-chlorophenyl)-2-[[(4-methylphenyl)imino]methyl]-3-chloro-benzenamine (2g).



Following procedure A, **1a** (0.396 g, 1.5 mmol) was used as substrate. The amount of 4-chloroaniline and NaOH were both 1.5 mmol. After 12h heating, product **2g** (0.437 g, 82%) was obtained.

¹H NMR (300 MHz, CDCl₃) δ 11.89 (s, 1H), 9.25 (s, 1H), 7.33-7.30 (m, 2H), 7.24-7.18 (m, 6H), 7.16-7.13 (m, 2H), 6.83 (dd, ${}^{3}J_{HH} = 5.4$, ${}^{4}J_{HH} = 3.3$ Hz, 1H), 2.38 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 159.12, 148.18, 147.85, 139.29, 138.36, 136.40, 132.15, 129.96, 129.48, 128.72, 123.96, 121.14, 118.76, 114.79, 112.00, 21.07. HRMS-ESI (m/z) Calcd for C₂₀H₁₆Cl₂N₂ [M+H]: 355.0769; Found: 355.0770.

N-naphthyl-2-[[(4-methylphenyl)imino]methyl]-3-chloro-benzenamine (2h)



Following procedure A, **1a** (0.396 g, 1.5 mmol) was used as substrate. The amount of naphthyl amine and NaOH were both 1.5 mmol. After 12h heating, product **2h** (0.511g, 92%) was obtained.

¹H NMR (300 MHz, CDCl₃) δ 12.31 (s, 1H), 9.38 (s, 1H), 8.20-8.17 (m, 1H), 8.91-8.88 (m, 1H), 7.70 (d, ${}^{3}J_{\text{HH}} = 7.8$ Hz, 1H), 7.58 (d, ${}^{3}J_{\text{HH}} = 7.2$ Hz, 1H), 7.53-7.48 (m, 3H), 7.21 (s, 4H), 7.10-7.00 (m, 2H), 6.80 (dd, ${}^{3}J_{\text{HH}} = 7.2$, ${}^{4}J_{\text{HH}} = 1.5$ Hz, 1H), 2.37 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 159.21, 149.46, 148.24, 138.18, 136.73, 136.28, 134.82, 132.14, 129.95, 129.58, 128.39, 126.34, 126.32, 125.78, 124.96, 122.80, 121.23, 120.43, 118.08, 114.60, 112.43, 21.04. HRMS-ESI (m/z) Calcd for C₂₄H₁₉ClN₂ [M+H]: 371.1315; Found: 371.1286.

N-isopropyl-2-(4-fluorophenyl)amino-6-chloro-benzenemethanamine (2i).



Following procedure A, **1g** (0.324 g, 1.5 mmol) was used as substrate. The amount of 4-fluoroaniline and NaOH were both 1.5 mmol. After 12h heating, it was handled following procedure B. Product **2i** (0.351 g, 80%) was

obtained.

¹H NMR (300 MHz, CDCl₃) δ 8.83 (s, 1H), 7.09-7.04 (m, 2H), 7.01-6.97(m, 4H), 6.83(dd, ³*J*_{HH} = 7.8, ⁴*J*_{HH} = 1.5 Hz, 1H), 4.06 (s, 2H), 2.86(m, 1H), 1.15 (d, ³*J*_{HH} = 6.3 Hz, 6H), 0.89 (m, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 157.81(d, ¹*J*_{CF} = 238.3Hz), 146.46, 138.88 (d, ⁴*J*_{CF} = 2.3 Hz), 134.46, 128.42, 123.75, 120.55 (d, ³*J*_{CF} = 7.7 Hz), 120.22, 115.92 (d, ²*J*_{CF} = 22.3 Hz), 112.66, 48.43, 45.79, 23.09. ¹⁹F NMR (376 MHz, CDCl₃) δ 122.36-122.43(m, 1F). HRMS-ESI (m/z) Calcd for C₁₆H₁₈ClFN₂ [M+H]: 293.1221; Found: 293.2237.

N-nomalbutyl-2-(4-methylphenyl)amino-6-chloro-benzenemethanamine (2j).



Following procedure A, **1h** (0.345 g, 1.5 mmol) was used as substrate. The amount of 4-methylaniline and NaOH were both 1.5 mmol. After 12h heating, it was handled following procedure B. Product 2j (0.284 g, 63%) was obtained.

¹H NMR (300 MHz, CDCl₃) δ 8.72 (s, 1H), 7.15 (dd, ³*J*_{HH} = 8.1, ⁴*J*_{HH} = 0.9 Hz, 1H), 7.10-6.96 (m, 5H), 6.81 (dd, ³*J*_{HH} = 7.8, ⁴*J*_{HH} = 1.1 Hz, 1H), 4.05 (s, 2H), 2.68 (t, ³*J*_{HH} = 6.9 Hz, 2H), 2.31 (s, 3H), 1.57-1.48 (m, 2H), 1.47-1.34 (m, 2H), 1.18 (s, 1H), 0.93 (t, ³*J*_{HH} = 7.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 146.54, 140.19, 134.50, 130.65, 129.85, 128.36, 123.57, 119.89, 119.25, 112.96, 48.71, 48.18, 32.23, 20.72, 20.48, 13.99. HRMS-ESI (m/z) Calcd for C₁₈H₂₃ClN₂ [M+H]: 303.1628; Found: 303.3047.

N-cyclohexyl-2-(4-methylphenyl)amino-6-chloro-benzenemethanamine (2k).



Following procedure A, **1i** (0.384 g, 1.5 mmol) was used as substrate. The amount of 4-methylaniline and NaOH were both 1.5 mmol. After 12h heating, it was handled following procedure B. Product **2k** (0.402g, 82%) was obtained.

¹H NMR (300 MHz, CDCl₃) δ 8.67 (s, 1H), 7.30 (dd, ³*J*_{HH} = 8.1, ⁴*J*_{HH} = 0.9 Hz, 1H), 7.17-7.06 (m, 3H), 7.00 (dt, ³*J*_{HH} = 8.4, ⁴*J*_{HH} = 2.1 Hz, 2H), 6.75 (td, ³*J*_{HH} = 7.5, ⁴*J*_{HH} = 1.2 Hz, 1H), 3.84 (s, 2H), 2.55-2.46 (m, 1H), 2.30 (s, 3H), 1.98-1.94 (m, 2H), 1.76-1.71 (m, 2H), 1.63-1.58 (m, 2H), 1.32-1.27 (m, 2H), 1.19-1.12 (m, 2H), 0.91 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 144.43, 140.79, 129.91, 129.80, 129.75, 128.04, 126.85, 118.93, 118.38, 114.58, 56.09, 50.58, 33.59, 26.21, 24.88, 20.70. HRMS-ESI (m/z) Calcd for C₂₀H₂₅ClN₂ [M+H]: 329.1785; Found: 329.1722.

N-isopropyl-2-(4-methylphenyl)amino-6-chloro-benzenemethanamine (21).



Following procedure A, **1g** (0.324 g, 1.5 mmol) was used as substrate. The amount of 4-methylaniline and NaOH were both 1.5 mmol. After 12h heating, it was handled following procedure B. Product **2l** (0.370 g, 86%) was obtained.

¹H NMR (300 MHz, CDCl₃) δ 8.76 (s, 1H), 7.18-6.96 (m, 6H), 6.82 (d, ³*J*_{HH} = 7.8 Hz, 1H), 4.05 (s, 2H), 2.91-2.83 (m, 1H), 2.31 (s, 3H), 1.15 (d, ³*J*_{HH} = 6.3 Hz, 6H), 1.02 (s, 1H). ¹³C NMR (75 MHz, CDCl₃) δ 146.44, 140.29, 134.42, 130.55, 129.93, 128.40, 123.85, 120.01, 119.03, 113.05, 48.50, 45.87, 23.17, 20.78. HRMS-ESI (m/z) Calcd

for C₁₇H₂₁ClN₂ [M+H]:289.1472; Found: 289.3189.

N-(4-methylphenyl)-2-[(naphthylimino)methyl]-3-chloro-benzenamine (2m).



Following procedure A, **1e** (0.450 g, 1.5 mmol) was used as substrate. The amount of 4-methylaniline and NaOH were both 1.5 mmol. After 12h heating, product **2m** (0.550 g, 99%) was obtained.

¹H NMR (300 MHz, CDCl₃) δ 11.91 (s, 1H), 9.35 (s, 1H), 8.28 (dd, ${}^{3}J_{HH} = 6.9$, ${}^{4}J_{HH} = 2.7$ Hz, 1H), 7.87, (dd, ${}^{3}J_{HH} = 7.2$, ${}^{4}J_{HH} = 2.4$ Hz, 1H), 7.75 (d, ${}^{3}J_{HH} = 8.4$ Hz, 1H), 7.55-7.48 (m, 3H), 7.25-7.12 (m, 7H), 6.81 (dd, ${}^{3}J_{HH} = 7.2$, ${}^{4}J_{HH} = 1.5$ Hz, 1H), 2.36 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 160.39, 149.05, 148.55, 138.44, 137.72, 133.97, 133.89, 132.47, 130.05, 128.54, 127.88, 126.44, 126.21, 126.17, 123.43, 117.95, 114.51, 113.86, 112.02, 20.93. HRMS-ESI (m/z) Calcd for C₂₄H₁₉ClN₂ [M+H]:371.1315; Found: 371.1286.

N-(4-methylphenyl)-2-[[(2-chlorophenyl)imino]methyl]-3-chloro-benzenamine (2n).



Following procedure A, **1b** (0.427 g, 1.5 mmol) was used as substrate. The amount of 4-methylaniline and NaOH were both 1.5 mmol. After 12h heating, product **2n** (0.368 g, 69%) was obtained.

¹H NMR (300 MHz, CDCl₃) δ 11.94 (s, 1H), 9.29 (s, 1H), 7.47 (dd, ³*J*_{HH} = 7.8, ⁴*J*_{HH} = 1.4 Hz, 1H), 7.36-7.28 (m, 2H), 7.24-7.09 (m, 7H), 6.77 (dd, ³*J*_{HH} = 7.2, ⁴*J*_{HH} = 1.4 Hz, 1H), 2.35 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 159.97, 149.14, 147.48, 138.40, 137.73, 133.78, 132.67, 129.96, 129.94, 129.83, 127.81, 127.05, 123.44, 119.21, 117.71, 114.05, 112.07, 20.93. HRMS-ESI (m/z) Calcd for C₂₀H₁₆Cl₂N₂ [M+H]:355.0769; Found: 355.0770.

N-(4-methylphenyl)-2-[[(4-methylphenyl)imino]methyl]-3-(4-methylphenyl)amino -benzenamine (**20**).

Following procedure A, **1a** (0.396 g, 1.5 mmol) was used ⁸

as substrate. The amount of 4-methylaniline and NaOH were both 3.0 mmol. After 24h heating, product 20 (0.395 g, 65%) is obtained.

¹H NMR (300 MHz, CDCl₃) δ 8.95 (s, 1H), 8.54 (s, 2H), 7.15-7.09 (m, 7H), 7.04-6.98 (m, 6H), 6.74 (d, ${}^{3}J_{HH} = 8.1$ Hz, 2H), 2.33 (s, 3H), 2.31 (s, 6H). ${}^{13}C$ NMR (75 MHz, CDCl₃) & 158.63, 149.58, 147.42, 141.01, 135.88, 133.19, 132.02, 130.30, 130.18, 121.47, 120.84, 111.52, 109.27, 21.38, 21.18. HRMS-ESI (m/z) Calcd for $C_{28}H_{27}N_3$ [M+H]: 406.2283; Found: 406.2242.

N-(4-methylphenyl)-2-(4-methylphenyl)amino-benzenemethanamine (2p).



Following procedure A, 1f (0.344 g, 1.5 mmol) was used as substrate. The amount of 4-methylaniline and NaOH were both 1.5 mmol. After 12h/24h heating, it was handled following procedure B. Product 2p [0.076 g, 17%

(12h) / 0.315 g, 70% (24h)] was obtained.

¹H NMR (300 MHz, CDCl₃) δ 7.33 (s, 1H), 7.29 (d, ³*J*_{HH} = 5.7 Hz, 1H), 7.22 (t, ³*J*_{HH} = 7.8 Hz, 1H), 7.07 (dd, ${}^{3}J_{HH}$ = 8.4, ${}^{4}J_{HH}$ = 2.7 Hz, 4H), 6.96 (d, ${}^{3}J_{HH}$ = 8.4 Hz, 2H), 6.89-6.85 (m, 2H), 6.72 (d, ${}^{3}J_{HH} = 8.4$ Hz, 2H), 4.26 (s, 2H), 3.59 (s, 1H), 2.30 (s, 3H), 2.29 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 145.98, 143.79, 140.38, 130.67, 130.44, 129.88, 129.86, 128.75, 128.29, 126.26, 120.00, 119.13, 116.09, 114.41, 48.25, 20.72, 20.50. HRMS-ESI (m/z) Calcd for C₂₁H₂₂N₂ [M+H]: 303.1861; Found: 303.1867.

N-(4-methyphenyl)-perchloroaniline (2q).



Following procedure A, hexachlorobenzene (0.428 g, 1.5 mmol) was used as substrate. The amount of 4-methylaniline and NaOH were both 1.5 mmol. After 12h heating, the crude product was purified by column chromatography using

petroleum ether as eluent. Pure product 2q (0.304 g, 57%) is obtained.

¹H NMR (300 MHz, CDCl₃) δ 7.07 (d, ³J_{HH} = 8.1 Hz, 2H), 6.65 (d, ³J_{HH} = 8.1 Hz, 2H), 5.95 (s, 1H), 2.30 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 139.61, 138.05, 132.06, 131.93, 129.69, 127.86, 127.58, 117.84, 20.69. HRMS-ESI (m/z) Calcd for 9

C₁₃H₈Cl₅N [M+H]: 355.9148; Found: 355.9078.

¹H NMR of 2a











¹³C NMR of **2b**





¹³C NMR of **2c**



¹H NMR of **2d**



¹³C NMR of **2d**









¹H NMR of $\mathbf{2f}$



```
<sup>13</sup>C NMR of 2f
```





```
<sup>13</sup>C NMR of 2g
```

-11.89









¹H NMR of **2i**







¹⁹F NMR of **2i**



¹H NMR of **2j**





¹H NMR of **2k**



¹³C NMR of **2k**



¹H NMR of **2**I



 $^{13}\mathrm{C}\,\mathrm{NMR}$ of $\mathbf{2I}$



¹H NMR of **2m**



¹³C NMR of **2m**



23



¹H NMR of **2n**



¹³C NMR of **2n**



24



¹H NMR of **20**





¹H NMR of **2p**



¹³C NMR of **2p**



¹H NMR of 2q



¹³C NMR of **2q**

