Supporting Information

Reductive and Transition-Metal-Free: Oxidation of Secondary Alcohols by Sodium Hydride

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Table of Contents:

Part I: General Information	S1
Part II: General Procedures	S2-S3
Part III: Characterization Data for the Synthesized Compounds	S3-S11
Part IV: ^1H and ^{13}C NMR Spectra for the Synthesized Compounds	S12-S77

Part I: General Information

Reagents were purchased at the highest commercial quality and used without further purification unless otherwise noted. All reactions were carried out in glassware under a nitrogen atmosphere with dry solvents. Sodium hydride was purchased from Acros, 60% dispersion in mineral oil in soluble bags, in resealable cans. Anhydrous THF was distilled from sodium-benzophenone. Yields refer to chromatographically purified products unless otherwise stated. Silica gel (ZCX-II, 200-300 mesh) used for flash column chromatography was purchased from Qing Dao Hai Yang Chemical Industry Co. of China. ^1H -NMR and ^{13}C -NMR spectra were recorded on either a Brüker Advance 300 (^1H : 300 MHz, ^{13}C : 75 MHz), or Brüker Advance 500 (^1H : 500 MHz, ^{13}C : 125 MHz). Mass spectrometric data were obtained using ABI-Q Star Elite high resolution mass spectrometer. The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, b = broad.

Part II: General Procedures

Oxidation of secondary alcohols using NaH

To a round-bottom flask that was flame-dried and cooled under nitrogen was added alcohol (1.0 mmol) and THF (5 mL). After stirring at 0 °C for 5 min, sodium hydride (2.0 mmol, 2.0 eq) was added as solid in one portion. The reaction mixture was then allowed to warm to room temperature and stirred for the indicated time [Note: at this point NaH may be recovered: the reaction mixture was allowed to stand for 20 min, organic layer was then separated from settled NaH precipitate by transferring into another flask under N₂ stream. The precipitate was washed three times with THF, dried under *vacuum*, and stored for re-use]. The reaction was quenched by addition of saturated ammonium chloride solution (5 mL), extracted with EtOAc (15 mL×2), washed by brine (15 mL), and the organic layer was dried over Mg₂SO₄ and concentrated under *vacuum*. The crude product was purified by flash chromatography on silica gel to give the desired compound.

Preparation of amides using NaH

To a round-bottom flask that was flame-dried and cooled under nitrogen was added THF (5 mL), aldehyde (1.0 mmol) and secondary amine (4.0 mmol, 4.0 eq). After stirring for one hour at room temperature, sodium hydride (2mmol, 2.0 eq, or the amount as specified in Scheme 6 in the text) was added in one portion (or multiple portions as noted). Caution: gas evolved! The reaction mixture was stirred for the indicated time, quenched by addition of saturated ammonium chloride solution (5 mL), extracted with EtOAc (15 mL×2), and washed with brine (15 mL). The combined organic phase was dried over Mg₂SO₄ and concentrated under *vacuum*. The crude product was purified by flash chromatography on silica gel to give the desired compound.

Preparation of amide using NaHMDS or KHMDS

To a round-bottom flask that was flame-dried and cooled under nitrogen was added THF (7 mL), picolinaldehyde (0.1 mL, 1.05 mmol, 1.0 eq), and diethylamine (0.44 mL, 4.2 mmol, 4.0 eq). After stirring at room temperature for 5 minutes, the reaction mixture was cooled to -78 °C and after another ten minutes, NaHMDS (2.0 M in THF, 1.6mL, 3.2 mmol, 3.0 eq) or KHMDS (0.5 M in toluene, 3.0 eq) was added dropwise *via* syringe. The reaction was then allowed to warm to room temperature and stirred for 10hs. The mixture was

quenched by addition of saturated ammonium solution (5 mL), extracted with EtOAc (15 mL $\times$ 2) and washed with brine (15 mL). The combined organic phase was dried over Mg_2SO_4 and concentrated under *vacuum*. The crude product was purified by flash chromatography on silica gel to give the desired compound.

When NaHMDS was used, the desired amide was isolated in 39% yield together with 50% of the reduction product pyridine-2-ylmethanol. When KHMDS was used, the desired amide was isolated almost quantitatively, in 97% yield.

Part III: Characterization Data for the Synthesized Compounds

Compound **2** was obtained in 95% yield (eluent: *n*-hexane/EtOAc = 1:5, R_f = 0.5) from the reaction of the corresponding alcohol (prepared by following the literature procedure^[1]) with NaH in THF for 10 hs. ^1H NMR (500 MHz, CDCl_3) δ 3.06 (s, 6H), 3.86 (s, 3H), 6.60-6.62 (dd, J = 5.8 Hz, 2.6, 1H), 6.93-6.95 (d, J = 8.7 Hz, 1H), 7.18-7.19 (d, J = 2.5 Hz, 2H), 7.26 (s, 1H), 8.09-8.11 (d, J = 8.8 Hz, 2H), 8.30-8.31 (d, J = 5.8 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 39.15, 55.40, 107.18, 108.08, 113.36, 129.59, 133.41, 148.56, 154.96, 156.21, 163.35, 193.53. Anal. Calcd for $\text{C}_{15}\text{H}_{16}\text{N}_2\text{O}_2$: C, 70.29; H, 6.29. Found: C, 70.11; H, 6.34.

Compound **5** was obtained in 99% yield (eluent: *n*-hexane/EtOAc = 1:1, R_f = 0.8) after 23 hs. ^1H NMR (500 MHz, CDCl_3) δ 3.08 (s, 6H), 6.68-6.70 (d, J = 9.1 Hz, 2H), 7.41-7.43 (m, 1H), 7.83-7.87 (m, 1H), 7.91-7.93 (d, J = 7.8 Hz, 1H), 8.06-8.08 (d, J = 9.1 Hz, 2H), 8.69-8.70 (dd, J = 2.4 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 40.00, 110.55, 123.63, 124.29, 125.16, 133.43, 136.76, 148.20, 153.52, 156.84, 191.69. [T. Högberg, B. Ulff, *J. Med. Chem.* **1981**, 24, 1499]

Compound **6** was obtained in 94% yield (eluent: *n*-hexane/EtOAc = 4:1, R_f = 0.2) after 16 hs. ^1H NMR (500 MHz, CDCl_3): δ 3.88 (s, 3H), 6.96-6.98 (d, J = 8.9, 2H), 7.44-7.47 (m, 1H), 7.86-7.89 (m, 1H), 7.98-7.99 (d, J = 7.8 Hz, 1H), 8.12-8.14 (d, J = 8.8 Hz, 2H), 8.70-8.71 (dd, J = 2.3 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 55.42, 113.50, 124.42, 125.70, 129.06, 133.46, 136.89, 148.31, 155.86, 163.57, 192.12. [M. Froimowitz, Y. Gu, L. A. Dakin, P. M.

Nagafuji, C. J. Kelley, D. Parrish, J. R. Deschamps, A. Janowsky. *J. Med. Chem.* **2007**, 50, 219]

Compound **7** was obtained in 88 % yield (eluent: *n*-hexane/EtOAc = 1:1, R_f = 0.8) after 16 hs. ^1H NMR (500MHz, CDCl_3) δ 0.95-0.98 (t, J = 7.4 Hz, 3H), 1.40-1.45(m, 2H), 1.74-1.80 (m, 2H), 2.86-2.89 (t, J = 7.8 Hz, 2H), 3.09 (s, 6H), 6.68-6.70 (d, J = 9.0 Hz, 2H), 7.25-7.26 (d, J = 8.1 Hz, 1H), 7.79-7.81 (d, J = 9.0 Hz, 2H), 7.96-7.98(dd, J = 8.0, 2.2 Hz, 1H), 8.85-8.86 (d, J = 1.8 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 13.87, 22.47, 31.80, 38.20, 39.99, 110.74, 122.14, 124.58, 132.24, 132.60, 127.20, 150.04, 153.60, 165.31, 192.82. HRMS (ESI) exact mass calcd for $[\text{M}+\text{H}]^+$ 283.1805, found 283.1812.

Compound **8** was obtained in 76% yield (eluent: *n*-hexane/EtOAc = 4:1, R_f = 0.6) after 14 hs. ^1H NMR (300 MHz, CDCl_3): δ 2.70 (s, 3H), 7.42-7.46 (m, 1H), 7.78-7.83 (m, 1H), 7.99-8.02 (dd, J = 7.8, 0.8, 1H), 8.65-8.66 (d, J = 3.0, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ 25.72, 121.56, 127.02, 136.76, 148.90, 153.47, 200.04. [M. Uyanik, M. Akakura, K. Ishihara, *J. Am. Chem. Soc.* **2009**, 131, 251]

Compound **9** was obtained in 81% yield (eluent: *n*-hexane/EtOAc = 4:1, R_f = 0.4) from the reaction of the corresponding alcohol (prepared by the reaction of picolinaldehyde with cyclopropylmagnesium chloride) with NaH in THF for 17 hs. ^1H NMR (500 MHz, CDCl_3) δ 1.06-1.10 (m, 2H), 1.21-1.24 (m, 2H), 3.45-3.50 (m, 1H), 7.43-7.46 (dd, 1H), 7.79-7.82 (m, 1H), 7.99-8.01 (d, J = 7.8 Hz, 1H), 8.69-8.71 (d, J = 4.4 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 12.51, 15.67, 121.63, 126.78, 136.75, 148.89, 153.73, 201.29. [*J. Heterocyclic Chem.* **1975**, 12, 413]

Compound **10** was obtained in 99% yield (eluent: *n*-hexane/EtOAc = 4:1, R_f = 0.8) after 20 hs. ^1H NMR (300 MHz, CDCl_3) δ 2.57 (s, 3H), 7.11-7.14 (t, J = 4.2 Hz, 1H), 7.63-7.64 (d, J = 4.2 Hz, 1H), 7.69-7.70 (d, J = 3.6 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 26.88, 128.07,

132.44, 133.75, 144.51, 190.72. [C. R. Graves, B-S. Zeng, S. T. Nguyen, *J. Am. Chem. Soc.* **2006**, 128, 12596]

Compound **11** was obtained in 24% yield (eluent: *n*-hexane/EtOAc = 4:1, R_f = 0.4) after 19 hs (this low isolated yield was also in part due to its instability on silica gel). ^1H NMR (500 MHz, CDCl_3): δ 2.49 (s, 3H), 6.53-6.54 (dd, J = 3.5, 1.6 Hz, 1H), 7.18-7.19 (d, J = 3.4 Hz, 1H), 7.58-7.59 (d, J = 0.8 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 25.96, 112.20, 116.97, 146.29, 153.07, 186.69. [*Synth. Commun.* **2008**, 38, 255]

Compound **12** was obtained in 59% yield (eluent: *n*-hexane/EtOAc = 6:1, R_f = 0.8) after 16 hs. Note that the GC yield of this reaction was 75%, some product was lost during purification due to its volatility. ^1H NMR (300 MHz, CDCl_3) δ 2.61 (s, 3H), 7.44-7.49 (m, 2H), 7.54-7.59 (m, 1H), 7.95-7.98 (t, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 26.58, 128.26, 128.52, 133.07, 137.05, 198.16. [I, Pérez, J. P. Sestelo, L. A. Sarandeses, *J. Am. Chem. Soc.* **2001**, 123, 4155]

Compound **13** was obtained in 86% yield (eluent: *n*-hexane/EtOAc = 4:1, R_f = 0.7) after 16 hs. ^1H NMR (300 MHz, CDCl_3) δ 2.58 (s, 3H), 7.42-7.44 (d, J = 8.4, 2H), 7.88-7.90 (d, J = 8.4 Hz, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 26.50, 128.84, 129.68, 135.37, 139.51, 196.76. [A. Leyva, A. Corma, *J. Org. Chem.* **2009**, 74, 2067]

Compound **14** was obtained in 87% yield (eluent: *n*-hexane/EtOAc = 4:1, R_f = 0.4) after 21 hs. ^1H NMR (500MHz, CDCl_3) δ 2.51 (s, 3H), 3.82(s,3H), 6.88-6.89 (d, J = 8.8 Hz, 2H), 7.88-7.90 (d, J = 8.8 Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 26.11, 55.27, 113.52, 130.18, 130.40, 163.34, 196.52. [*Synth. Commun.* **2008**, 38, 4188]

Compound **15** was obtained in 12% yield (eluent: *n*-hexane/EtOAc = 4:1, R_f = 0.4) after 30 hs. ^1H NMR (300 MHz, CDCl_3) δ 2.69 (s, 3H), 8.11-8.14 (d, J = 8.4 Hz, 2H), 8.32-8.34 (d, J = 8.4 Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 26.86, 123.83, 129.27, 141.48, 150.43, 196.14. [S. Manaam, S. K. Alamsetti, G. Sekar, *Adv. Synth. Catal.* **2007**, 349, 2253]

Compound **16** was obtained in 53% (eluent: *n*-hexane/EtOAc = 16:1, R_f = 0.8) after 18 hs. ^1H NMR (500 MHz, CDCl_3) δ 2.11-2.16 (m, 2H), 2.64-2.66 (t, 2H), 2.95-2.97 (t, 2H), 7.23-7.31 (m, 2H), 7.44-7.47 (q, 1H), 8.02-8.03 (d, J = 7.6 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 23.23, 29.65, 39.10, 126.55, 127.10, 128.69, 132.59, 133.29, 144.40, 198.17. [T. Mahammad, A. Shaikh, A. Sudalai, *Eur. J. Org. Chem.* **2008**, 29, 4877]

Compound **17** was obtained in 90% yield (eluent: *n*-hexane/EtOAc = 16:1, R_f = 0.6) after 19hs. ^1H NMR (300 MHz, CDCl_3) δ 7.45-7.49 (t, J = 7.5, 4H), 7.55-7.60 (t, J = 7.2 Hz, 2H), 7.79-7.81 (d, J = 7.5 Hz, 4H); ^{13}C NMR (75 MHz, CDCl_3) δ 128.17, 129.95, 132.33, 137.43, 196.65. [J. Srogl, S. Voltrova, *Org. Lett.* **2009**, 11, 843]

Compound **18** obtained in 62% yield (eluent: *n*-hexane/EtOAc = 16:1, R_f = 0.4) after 20 hs. ^1H NMR (500 MHz, CDCl_3) δ 2.73 (s, 3H), 7.55-7.63 (m, 2H), 7.88-7.91 (t, 2H), 7.96-7.98 (d, J = 3.0 Hz, 1H), 8.03-8.05 (dd, J = 8.6, 1.6 Hz, 1H), 8.47 (s, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 26.57, 123.92, 126.74, 127.77, 128.39, 128.41, 129.53, 130.10, 132.57, 134.62, 135.62, 197.92. [Z. Hyder, J. Ruan, J. Xiao, *Chem. Eur. J.* **2008**, 14, 5555]

Compound **19** was obtained in 40% yield (eluent: *n*-hexane/EtOAc = 1:1, R_f = 0.8) from the reaction of the corresponding alcohol (prepared by the reaction of 1-methyl-imidazol and

trans-2-hexenal referred to the literature procedure ^[2]) with NaH in THF for 36 hs. ¹H NMR (500 MHz, CDCl₃) δ 0.96-0.99 (m, 3H), 2.03-2.15 (m, 2H), 3.81-3.82 (m, 2H), 3.98(s, 3H), 5.63-5.66 (m, 2H), 7.01(s, 1H), 7.13(s, 1H); ¹³C NMR(125 MHz, CDCl₃) δ 13.45, 13.93, 20.91, 25.60, 36.04, 37.64, 42.71, 120.44, 121.04, 126.87, 129.05, 129.07, 135.27, 136.52, 142.89, 190.73, 191.25. Anal. Calcd for C₁₀H₁₄N₂O: C, 67.39; H, 7.92. Found: C, 67.69; H, 8.01.

Compound **22** was obtained in 54% yield (eluent: *n*-hexane/EtOAc = 4:1, *R*_f = 0.9) from the reaction of the corresponding allylic alcohol (*E*)-3-phenyl-1-(thiazol-2-yl)-prop-2-en-1-ol (prepared by the reaction of 2-bromothiazole and *trans*-cinnamaldehyde following the literature procedure ^[2]) with NaH in THF for 9 hs. ¹H NMR (500 MHz, CDCl₃) δ 3.08-3.11 (t, *J* = 7.7 Hz, 2H), 3.49-3.52 (t, *J* = 7.7 Hz, 2H), 7.18-7.21 (m, 1H), 7.25-7.32(m, 4H), 7.65-7.66 (d, *J* = 3.0 Hz, 1H), 7.98-7.99 (d, *J* = 3.0 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 29.83, 40.08, 126.10, 126.16, 128.43, 128.46, 140.73, 144.69, 167.00, 192.93. Anal. Calcd for C₁₂H₁₁NOS: C, 66.33; H, 5.10. Found: C, 66.72; H, 5.42.

Compound **23** was obtained in 64% yield (eluent: *n*-hexane/EtOAc = 16:1, *R*_f = 0.8) from the reaction of the corresponding allylic alcohol 1-(thiazol-2-yl)-prop-2-en-1-ol (prepared by the reaction of 2-bromothiazole and arcolein following the literature procedure ^[2]) with NaH in THF for 9 hs. ¹H NMR (300 MHz, CDCl₃) δ 1.21-1.26 (t, *J* = 7.4 Hz, 3H), 3.14-3.21 (q, *J* = 7.4 Hz, 2H), 7.65-7.66 (d, *J* = 2.9 Hz, 1H), 7.97-7.98 (d, *J* = 2.9 Hz, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 7.83, 31.84, 125.93, 144.56, 166.99, 194.46. HRMS (ESI) exact mass calcd for [M+H]⁺ 142.0321, found 142.0330.

Compound **24** was obtained in 68% yield (eluent: *n*-hexane/EtOAc = 16:1, *R*_f = 0.8) from the reaction of the corresponding allylic alcohol (*E*)-1-(thiazol-2-yl)-hex-2-en-1-ol (prepared by the reaction of 2-bromothiazole and *trans*-hexenal following the literature procedure ^[2]) with NaH in THF for 9 hs. ¹H NMR (300 MHz, CDCl₃) δ 0.89-0.93 (q, 3H), 1.36-1.38 (m, 4H), 1.75-1.80 (m, 2H), 3.13-3.18 (t, *J* = 7.5 Hz, 2H), 7.66-7.67 (d, *J* = 3.0 Hz, 1H), 8.00-8.01 (d, *J* = 3.0 Hz, 1H); ¹³C NMR(75 MHz, CDCl₃) δ 13.91, 22.42, 23.72, 31.37, 38.47, 126.05, 144.62, 167.36, 194.20. [A. Dondoni, G. Fantin, M. Fogagnolo, A. Medici, P. Pedrini, *J. Org.*

Chem. **1988**, 53, 1748]

Compound **25** was obtained in 86% yield (eluent: *n*-hexane/EtOAc = 4:1, R_f = 0.9) from the reaction of the corresponding allylic alcohol 1-(4-methylthiazol-2-yl)- prop-2-en-1-ol (prepared by the reaction of 4-methylthiazole-2-carbaldehyde and vinyl magnesium bromide) with NaH in THF for 2 hs. ^1H NMR (300 MHz, CDCl_3) δ 1.21-1.26 (t, J = 7.2 Hz, 3H), 2.52 (d, J = 0.6 Hz, 3H), 3.12-3.20 (q, J = 7.2 Hz, 2H), 7.22 (d, 0.6, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ 7.88, 17.20, 31.84, 121.03, 155.10, 166.05, 194.48. HRMS (ESI) exact mass calcd for $[\text{M}+\text{H}]^+$ 156.0478, found 156.0471.

Compound **26** was obtained in 68% yield (eluent: *n*-hexane/EtOAc = 1:1, R_f = 0.9) from the reaction of the corresponding allylic alcohol (*E*)-1-(1-methyl-1H-imidazol-2-yl)-3-phenyl prop-2-en-1-ol (prepared by the reaction of 1-methyl-1H-imidazole with *trans*-cinnamaldehyde following the literature procedure [2]) with NaH in THF for 9 hs. ^1H NMR (500 MHz, CDCl_3) δ 3.04-3.07 (t, J = 7.7 Hz, 2H), 3.47-3.50 (t, J = 7.7 Hz, 2H), 3.99 (s, 3H), 7.01 (s, 1H), 7.13 (s, 1H), 7.17-7.20 (m, 1H), 7.27-7.29 (m, 4H); ^{13}C NMR (125 MHz, CDCl_3) δ 29.97, 36.04, 40.36, 125.94, 126.80, 128.33, 128.42, 129.01, 141.08, 142.98, 192.01. Anal. Calcd for $\text{C}_{13}\text{H}_{14}\text{N}_2\text{O}$: C, 72.87; H, 6.59. Found: C, 72.76; H, 6.52.

Compound **27** was obtained in 68% yield (eluent: *n*-hexane/EtOAc = 4:1, R_f = 0.8) from the reaction of the corresponding allylic alcohol 1-(pyridin-2-yl)-prop-2-en-1-ol (prepared by the reaction of picolinaldehyde and vinyl magnesium bromide) with NaH in THF for 9 hs. ^1H NMR (300 MHz, CDCl_3) δ 1.16-1.21 (t, J = 6.9 Hz, 3H), 3.18-3.25 (q, J = 6.9 Hz, 2H), 7.42(s, 1H), 7.78-7.83 (t, J = 7.0 Hz, 1H), 8.00-8.02(d, J = 7.5 Hz, 1H), 8.64 (s, 1H). [J. Easmon, G. Pürstinger, K-S, Thies, G. Heinisch, J. Hofmann. *J. Med. Chem.* **2006**, 49, 6343]

Compound **28** was obtained in 30% yield (eluent: *n*-hexane/EtOAc = 4:1, R_f = 0.7) from the reaction of the corresponding allylic alcohol (*E*)-3-phenyl-1-(pyridin-2-yl)-prop-2-en-1-one (prepared by the reaction of 2-bromopyridine and *trans*-cinnamaldehyde following the literature procedure ^[2]) with NaH in THF for 8 hs. ¹H NMR (500 MHz, CDCl₃) δ 3.07-3.10 (t, J = 7.6 Hz, 2H), 3.57-3.58 (t, J = 7.6 Hz, 2H), 7.18-7.21 (dd, 1H), 7.29 (b, 4H), 7.45-7.47 (m, 1H), 7.81-7.85 (m, 1H), 8.05 (d, J = 7.8 Hz, 1H), 8.67-8.68 (d, J = 4.5 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 29.89, 39.36, 121.77, 125.94, 127.03, 128.37, 128.46, 136.81, 141.43, 148.93, 153.39, 200.96. [*J. Chem. Soc. Perkin. Trans.* **1987**, 4, 699]

Compound **29** was obtained in 50% yield (eluent: *n*-hexane/EtOAc = 4:1, R_f = 0.8) from the reaction of the corresponding allylic alcohol (*E*)-1-(pyridin-2-yl)-hex-2-en-1-ol (prepared by the reaction of 2-bromopyridine and *trans*-hexenal following the literature procedure ^[2]) with NaH in THF for 8 hs. ¹H NMR (500 MHz, CDCl₃) δ 0.89-0.92 (t, J = 6.2 Hz, 3H), 1.36-1.38 (m, 4H), 1.72-1.75 (m, 2H), 3.19-3.22 (t, J = 7.5 Hz, 2H), 7.44-7.46 (m, 1H), 7.80-7.84 (m, 1H), 8.02-8.04 (d, J = 7.8 Hz, 1H), 8.67-8.68 (d, J = 4.6 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 13.89, 22.50, 23.72, 31.55, 37.64, 121.73, 126.85, 136.66, 148.88, 153.72, 202.18. Anal. Calcd for C₁₁H₁₅NO: C, 74.54; H, 8.53. Found: C, 74.43; H, 8.27.

Compound **30** was obtained in 32% yield (eluent: *n*-hexane/EtOAc = 4:1, R_f = 0.7) together with compound **29**. ¹H NMR (500 MHz, CDCl₃) δ 0.81-0.85 (m, 6H), 1.18-1.33 (m, 8H), 1.54-1.67 (m, 1H), 1.85-1.92 (m, 1H), 2.62-2.63 (d, J = 5.0 Hz, 1H), 3.22-3.26 (q, 1H), 3.38-3.43 (q, 1H), 4.28-4.31 (q, 1H), 7.37-7.44 (m, 2H), 7.77-7.85 (m, 2H), 8.00-8.01 (d, J = 7.8 Hz, 1H), 8.05-8.06 (d, J = 7.8 Hz, 1H), 8.51-8.52 (d, J = 4.6 Hz, 1H), 8.62-8.63 (d, J = 4.6 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 13.86, 14.07, 20.32, 22.90, 27.80, 30.10, 34.08, 36.01, 40.13, 46.74, 121.79, 122.03, 126.55, 126.67, 136.61, 136.66, 148.69, 148.78, 153.94, 154.16, 201.77, 205.16. HRMS (ESI) exact mass calcd for [M+H]⁺ 353.2224, found 353.2213.

Compound **32** was obtained in 90% yield (eluent: *n*-hexane/EtOAc = 10:1, R_f = 0.5) after 18 hs. ^1H NMR (300 MHz, CDCl_3) δ 5.62 (s, 2H), 6.80 (s, 2H), 6.92-6.93 (d, J = 1.8 Hz, 2H), 7.00-7.07 (m, 4H), 7.26-7.29 (t, J = 5.1 Hz, 2H), 7.35-7.43 (dd, J = 13.8 Hz, 8.1, 4H); ^{13}C NMR (75 MHz, CDCl_3) δ 75.71, 75.78, 115.27, 115.56, 125.83, 125.87, 125.97, 126.48, 128.71, 128.82, 128.93, 136.77, 145.54, 145.69, 160.79, 160.84, 164.06, 164.11. HRMS (ESI) exact mass calcd for $[\text{M}+\text{Na}]^+$ 421.0503, found 421.0522.

Compound **34** was obtained in 25 % yield (eluent: *n*-hexane/EtOAc = 4:1, R_f = 0.3) by the reaction of 4-methyl-2-thiazolecarbox- aldehyde with pyrrolidine (dried over molecular sieve for 2 days) after 12 hs. ^1H NMR (300 MHz, CDCl_3) δ 1.88-2.01 (m, 4H), 2.48 (s, 3H), 3.65-3.70 (t, J = 6.8 Hz, 2H), 4.07-4.12 (t, J = 6.8 Hz, 2H), 7.06 (s, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 17.36, 23.70, 26.54, 47.49, 48.93, 118.43, 153.84, 159.08, 164.70. HRMS (ESI) exact mass calcd for $[\text{M}+\text{H}]^+$ 197.0743, found 197.0748.

Compound **36** was obtained in 18% yield (eluent: *n*-hexane/EtOAc = 2:1, R_f = 0.3, 34% based on the recovered starting material) by the reaction of picolinaldehyde with pyrrolidine (dried over molecular sieve for 2 days) after 23 hs. ^1H NMR (300 MHz, CDCl_3) δ 1.92-1.92 (m, 4H), 3.68-3.72 (m, 4H), 7.31-7.32 (d, J = 3.9 Hz, 1H), 7.78-7.80 (d, J = 6.0 Hz, 2H), 8.58 (s, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 23.90, 26.44, 46.68, 48.94, 123.64, 124.53, 136.69, 147.85, 154.39, 166.39. [Z. Guo, E. D. Dowdy, W-S. Li, R. Polniaszek, E. Delaney, *Tetrahedron. Lett.* **2001**, 42, 1843]

Compound **37** was obtained in 55 % yield (eluent: *n*-hexane/EtOAc = 4:1, R_f = 0.2) after 10 hs when NaH was added in five small portions (note that the product yield was 5% when NaH was added in one portion). ^1H NMR (500 MHz, CDCl_3) δ 1.11-1.13 (t, J = 7.1 Hz, 3H),

1.23-1.26 (t, $J = 7.1$ Hz, 3H), 3.34-3.38 (q, $J = 7.1$ Hz, 2H), 3.52-3.56 (q, $J = 7.1$ Hz, 2H), 7.30-7.33 (dd, 1H), 7.56-7.58 (d, $J = 7.8$ Hz, 1H), 7.76-7.79 (m, 1H), 8.58-8.59 (d, $J = 4.8$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 12.75, 14.17, 40.08, 43.08, 122.91, 123.88, 136.65, 148.20, 155.25, 168.44. [M. Alessi, A. L. Larkin, K. A. Ogilvie, L. A. Green, S. Lai, S. Lopez, V. Snieckus. *J. Org. Chem.* **2007**, 72, 1588]

References:

1. D. Cuperly, P. Gros, Y. Fort, *J. Org. Chem.* **2002**, 67, 238–241.
2. A. J. Boersma, B. L. Feringa, G. Roelfes, *Org. Lett.* **2007**, 9, 3647-3650.

NMR300
 sample name: wxb090308-py-DMAPh-ketone
 solvent: cdcl3

NAME wxb090308-py-DMAPh-ketone
 EXPNO 2
 PROCNO 1
 Date_ 20090310
 Time 1.21
 INSTRUM spect
 PROBRD 5 mm PULP, 13C
 PULPROG zgpg30
 TD 32768
 F2 32768
 SOLVENT DMSO
 NS 1000
 DS 0
 SWH 18115.941 Hz
 FIDRES 0.1552855 Hz
 AQ 0.9944468 sec
 RG 161.3
 PC 27.600 usec
 DM 6.00 usec
 DE 236.3 K
 TE 1.00000000 sec
 d11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 PL 9.90 usec
 P1 -1.10 dB
 SFO1 75.4756731 MHz

===== CHANNEL f2 =====
 CEPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 P1L2 15.80 dB
 SFO2 300.1312005 MHz
 SI 32768
 SF 75.4677523 MHz
 WDM EX
 WSR 0
 SSB 1.00 Hz
 LB 0
 GB 0
 PC 1.40

sample: wxb-090301-4M-pyph-one
 solvent: CDCl₃

Bruker 300M szpku
 sample name: wxb090303-py2-acetone
 solvent: cdcl3

2.700

NAME wxb090303-py2-acetone
 EXPNO 1
 PROCNO 1
 Date_ 20090304
 Time 19.10
 INSTRUM spect
 PROBRD 5 mm PABUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT cdcl3
 NS 8
 DS 0
 SWH 5995.204 Hz
 FIDRES 0.091480 Hz
 AQ 5.4657526 sec
 RG 80.6
 DW 83.400 usec
 DE 6.00 usec
 TE 294.7 K
 D1 1.0000000 sec
 TDO 1
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 14.00 usec
 PL1 0.00 dB
 SFO1 300.1322510 MHz
 SI 32768
 SF 300.1300042 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

3.043

NMR300
 sample name: wxb090303-py2-acetone
 solvent: cdcl3

```

NAME      wxb090303-py2-acetone
EXPNO     1
PROCNO    1
Date_     20090304
Time      19.12
INSTRUM   spect
PROBHD    5 mm PABUL 13C
PULPROG   zgpg30
TD         32768
SOLVENT   DMSO
NS         301
DS         0
SWH        18115.941 Hz
FIDRES     0.552855 Hz
AQ         0.9044468 sec
RG         1290.2
RW         27.600 usec
DE         6.00 usec
TE         294.7 K
D1         1.00000000 sec
d11        0.03000000 sec
TDO        1

===== CHANNEL f1 =====
NUC1       13C
P1         9.90 usec
PL1        -1.10 dB
SFO1       75.4756731 MHz

===== CHANNEL f2 =====
CF2PRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
P2         0.00 dB
PL2        15.00 dB
SFO2       300.1312005 MHz
SI         32768
SF         75.4677339 MHz
WDW        RM
SSB         0
GB         0
PC         1.40
  
```

Bruker 300M szpku
 sample name: wxb090304-1-acethihiphene
 solvent: cdcl3

7.703
 7.691
 7.643
 7.629
 7.268
 7.142
 7.126
 7.113

2.567

NAME wxb090304-1-acethihiphene
 EXPNO 1
 PROCNO 1
 Date_ 20090305
 Time 15.13
 INSTRUM spect
 PROBRD 5 mm PABUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 5935.204 Hz
 FIDRES 0.091480 Hz
 AQ 5.4657526 sec
 RG 128
 DW 83.400 usec
 DE 6.00 usec
 TE 294.7 K
 D1 1.00000000 sec
 ID0 1

CHANNEL f1
 NUC1 1H
 P1 14.00 usec
 PL1 0.00 dB
 SFO1 300.132510 MHz
 SI 32768
 SF 300.130041 MHz
 WDW EM
 SSB 0
 GB 0
 EC 0.30 Hz
 FC 1.00

NMR300
 sample name: wxb090304-1-acethiphenene
 solvent: cdcl3

NAME wxb090304-1-acethiphenene
 EXPNO 2
 PROCNO 1
 Date_ 20090305
 Time 15.20
 INSTRUM spect
 PROBHD 5 mm PABUL 13C
 PULPROG zgpg30
 TD 32768
 SOLVENT DMSO
 NS 815
 DS 0
 SMH 18115.941 Hz
 FIDRES 0.552855 Hz
 AQ 0.9044468 sec
 RG 1149.4
 DW 27.600 usec
 DE 6.00 usec
 TE 295.4 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.90 usec
 PL1 -1.10 dB
 SFO1 75.4756731 MHz

===== CHANNEL f2 =====
 GEOPRG2 waltz16
 NUC2 1H
 P2 80.00 usec
 PL2 0.00 dB
 PL12 15.80 dB
 SFO2 300.1312005 MHz
 SI 32768
 SF 75.4677538 MHz
 MTW 32
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

sample name: wxb090303-ph-acetone2
solvent: cdcl3

7.977
7.956
7.950
7.591
7.575
7.567
7.547
7.543
7.488
7.466
7.462
7.447
7.438
7.273
7.265

2.617
2.608

sample name: wxb090303-ph-acetone2
solvent: cdcl3

sample name:wxp09021/-4ClPhetnone solvent:cdcl3

7.904
7.976

7.443
7.415
7.269

2.584

sample name: wxb090217-4C1phenylacetone
solvent: cdcl3

sample: wxb-090312-afa-tetralone
 solvent: CDCl₃

8.0355
 8.0202
 7.4717
 7.4568
 7.4446
 7.4421
 7.3103
 7.2952
 7.2814
 7.2663
 7.2503
 7.2358

2.9723
 2.9605
 2.9492
 2.6627
 2.6583
 2.6485
 2.6370

2.1462
 2.1341
 2.1217
 2.1095

0.0815
 0.0017

zhengchao20090213

NAME zhengchao20090213
EXPNO 1
PROCNO 1
Date_ 20090213
Time 21.32
INSTRUM spect
PROBHD 5 mm PABUL 13C
PULPROG zgpg30
TD 32768
FID 32768
SOLVENT CDCl3
NS 8
DS 1
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.421924 sec
RG 384
RM 104.400 usec
DN 45.000 usec
TE 293.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 300.132510 MHz
SI 32768
SF 300.1300071 MHz
NUC2 RM
SSB 0
LB 0.30 Hz
GB 0
PC 0.10

zhengchao20090213

```

NAME zhengchao20090213
EXPNO 2
PROCNO 1
Date_ 20090213
Time 21.36
INSTRUM spect
PROBHD 5 mm PABUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 54
DS 0
SWH 18115.941 Hz
FIDRES 0.552855 Hz
AQ 0.904468 sec
RG 181
RG 181
DW 27.600 usec
DE 6.00 usec
TE 293.6 K
D1 1.00000000 sec
d11 0.03000000 sec
TD0 1
  
```

```

===== CHANNEL F1 =====
NUC1 13C
P1 9.90 usec
PL1 1.10 dB
SFO1 75.4756731 MHz
  
```

```

===== CHANNEL F2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 15.00 dB
PL12 15.00 dB
SFO2 300.13208 MHz
SI 32768
SD 75.4677604 MHz
SOLVENT CDCl3
NS 54
DS 0
SWH 1.00 Hz
TE 293.6 K
D1 1.00000000 sec
d11 0.03000000 sec
TD0 1
  
```

500M 1H NMR
 sample: wxd-090329-No4-naphthacetone
 solvent: CDCl₃

2.7342

500M 1HMR
 sample: wxb-090401-No3-thia-cina-ol
 solvent: CDCl₃

7.7478
 7.7417
 7.4040
 7.3892
 7.3208
 7.3141
 7.3077
 7.2917
 7.2635
 7.2526
 6.8304
 6.7987
 6.4617
 6.4484
 6.4301
 6.4169
 5.6793
 5.6663

3.8297

0.0002

Bruker 300M szpku
 sample name: wxb090403-No1-thiazolevinyl1-ketone
 solvent: cdcl3

7.980
 7.971
 7.657
 7.647
 7.268

3.208
 3.184
 3.160
 3.135

1.256
 1.232
 1.207

NAME wxb090403-No1-thiazolevinyl1-
 EXPNO 1
 PROCNO 1
 Date_ 20090403
 Time 14.58
 INSTRUM spect
 PROBD 5 mm PABUL13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SM 5995.204 Hz
 FIDRES 0.091480 Hz
 AQ 5.4657526 sec
 RG 90.5
 DM 83.400 usec
 DE 6.000 usec
 TE 293.2 K
 D1 1.00000000 sec
 TDO 1
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 14.00 usec
 PL 0.00 dB
 SFO1 300.1322510 MHz
 SI 32768
 SF 300.1300041 MHz
 ME 0
 M1 0
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

NMR300
 sample name: wxb090403-No1-thiazolevinyl-ketone
 solvent: cdcl3

NAME wxb090403-No1-thiazolevinyl
 EXPNO 2
 PROCNO 1
 Date_ 20090403
 Time 15.00
 INSTRUM 5 mm PABULI 13C
 PROBRD 500 MHz
 PULPROG zgpg30
 FIDRES 0.32448
 T1 2.00
 T2 0.10
 SOLVENT CDCl3
 NS 164
 DS 4
 SM 18115.941 Hz
 FIDRES 0.55285 Hz
 AQ 0.904468 sec
 RG 161.3
 TM 27.600 usec
 DE 6.00 usec
 TE 293.8 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 TDO 1

===== CHANNEL F1 =====
 NUCL 13C
 P1 9.90 usec
 PL1 -1.10 dB
 SFO1 75.4756731 MHz

===== CHANNEL F2 =====
 CPDPRG2 waltz16
 NUCL2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PUL2 15.80 dB
 SFO2 300.1312005 MHz
 SI 32768
 SF 75.467537 MHz
 WDW EM
 SSB 0
 GB 1.00 Hz
 PC 1.40

Bruker 300M szpku
 sample name:wxb090402-NO3-thiazolehexenol
 solvent:cdcl3

Current Data Parameters
 NAME wxb090402-NO3-thiazolehex
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20090402
 Time 21.38
 INSTRUM spect
 PROBRD 5 mm PABUL 13C
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 5995.204 Hz
 FIDRES 0.091480 Hz
 AQ 5.4657526 sec
 RG 90.5
 DW 83.400 usec
 DE 6.00 usec
 TE 294.3 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.00 usec
 PL 0.00 dB
 SFO1 300.1322510 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300026 MHz
 KW 64
 NSB 24
 LB 0
 GB 0
 PC 1.00

7.725
7.714

7.310
7.303
7.293

5.953
5.930
5.902
5.880
5.765
5.746
5.742
5.714
5.696
5.453
5.433

3.853

2.091
2.068
2.043
2.003

1.474
1.449
1.425
1.400

0.940
0.934
0.916
0.909
0.885

NMR300
 sample name: wbo090402-No3-thiazolehexenol
 solvent: cdc13

NAME wbo090402-No3-thiazolehexenol
 EXPNO 1
 PROCNO 2
 Date_ 20090402
 Time 21.40
 INSTRUM spect
 PROBD 5 mm PNU1, 13C
 PULPROG zgpg30
 TD 32768
 SOLVENT DMSO
 NS 162
 DS 0
 SWH 18115.941 Hz
 FIDRES 0.552855 Hz
 AQ 0.9049468 sec
 RG 161.3
 DW 27.600 usec
 DE 6.00 usec
 TE 294.5 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.30 usec
 PL1 -1.10 dB
 SFO1 75.4756731 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 15.80 dB
 SP02 300.1312005 MHz
 SI 32768
 SF 75.4677537 MHz
 MTN EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Bruker 300M szpku
 sample name: wxb090402-No5-thiazolehex-ketone
 solvent: cdc13

```

NAME      wxb090402-No5-thiazolehex-
EXPNO     1
PROCNO    1
Date_     20090403
Time      15.24
INSTRUM   spect
PROBHD    5 mm PABUL 13C
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         0
DS         0
SWH        5995.204 Hz
FIDRES     0.001480 Hz
AQ          5.4657526 sec
RG          203.2
BO          83.400 usec
DE          6.00 usec
TE          293.4 K
D1          1.00000000 sec
TD0         1

===== CHANNEL f1 =====
NUC1       1H
P1         14.00 usec
PL         0.00 dB
SFO1       300.1322510 MHz
SI         32768
SF         300.1300043 MHz
WDW         EM
SSB         0
GB         0.30 Hz
PC          1.00
  
```

NMR300
 sample name: wxb090402-No5-thiazolehex- ketone
 solvent: cdcl3

NAME: wxb090402-No5-thiazolehex-
 EXPNO: 2
 PROCNO: 1
 Date_: 20090403
 Time: 15.27
 INSTRUM: spect
 PROBHD: 5 mm BBDUL 13C
 PULPROG: zgpg30
 TD: 32768
 SOLVENT: DMSO
 NS: 1216
 DS: 4
 SWH: 18115.941 Hz
 FIDRES: 0.152845 Hz
 AQ: 0.904468 sec
 RG: 161.3
 DW: 27.600 usec
 DE: 6.00 usec
 TE: 293.6 K
 D1: 1.00000000 sec
 d11: 0.03000000 sec
 TD0: 1

===== CHANNEL F1 =====
 NUC1: 13C
 P1: 9.90 usec
 PL1: -1.10 dB
 SFO1: 75.4756731 MHz

===== CHANNEL F2 =====
 CPDPRG2: waltz16
 NUC2: 1H
 PCPD2: 80.00 usec
 PL2: 0.00 dB
 PL12: 15.80 dB
 SFO2: 300.1312005 MHz
 SI: 32768
 SF: 75.4677513 MHz
 WDM: 0
 SSB: 0
 LB: 1.00 Hz
 GB: 0
 PC: 1.40

Bruker 300M szpku
 sample name: wxb090329-No1-thiazo
 solvent: cdcl3

Current Data Parameters
 NAME: wxb090329-No1-thiazo
 EXPNO: 1
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20090329
 Time: 22.24
 INSTRUM: spect
 PROBHD: 5 mm PABUL1K
 PULPROG: zgpg30
 TD: 65536
 SOLVENT: CDCl3
 NS: 8
 DS: 0
 SWH: 5995.204 Hz
 FIDRES: 0.091480 Hz
 AQ: 5.465526 sec
 RG: 512
 DW: 83.400 usec
 DE: 6.00 usec
 TE: 294.7 K
 D1: 1.0000000 sec
 TDO: 1

===== CHANNEL f1 =====
 NUC1: 1H
 P1: 14.00 usec
 PL1: 0.00 dB
 SFO1: 300.132510 MHz
 F2 - Processing parameters
 SI: 32768
 SF: 300.130040 MHz
 WDW: EM
 SSB: 0
 LB: 0.30 Hz
 GB: 0
 PC: 1.00

NMR300
wxlb0903
vent:ca

```

NAME          web090529-Nb1-1-haze
EXPNO         2
PROCNO        1
Date_         20090529
Time          22.56
INSTRUM       spect
PROBHD        5 mm BBO D1.13C
PULPROG       zgpg30
TD            32768
SOLVENT       DMSO
NS            500
DS            0
SWH            18115.941 Hz
FIDRES        0.532855 Hz
AQ            0.904468 sec
RG            67.5
RIG           27.00 usec
DR            6.00 usec
TE            299.8 K
DE            1.00000000 sec
D11           0.03000000 sec
D10           1
===== CHANNEL f1 =====
NUC1          13C
P1            9.90 usec
PL1           -1.10 dB
SFO1          75.4756731 MHz
===== CHANNEL f2 =====
CEPRG2        waltz16
NUC2          1H
PCPD2         80.00 usec
P2           0.00 dB
PL2           15.80 dB
SFO2          300.1312005 MHz
SI            32768
SF            75.4677515 MHz
K1            0
K2            0
C1S1          1.00 Hz
C1S2          0
C1S3          0
C1S4          1.40
PC            0

```

sample name: wxb090212-Py-vinal solvent: cdcl3

8.643
8.024
7.999
7.825
7.801
7.778
7.433
7.268

3.245
3.226
3.202
3.179

2.033

1.211
1.187
1.164

sample name: wxb090212-Py-vlnal
solvent: cdcl3

sample: wxb-030324-No2-up
solvent: CDCl₃

Bruker 300M szpku
 sample name: wxb090322-No3-py-ol-hex
 solvent: cdcl3

Current Data Parameters
 NAME wxb090322-No3-py-ol-hex
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20090324
 Time 0.26
 INSTRUM spect
 PROBD 5 mm PADUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 5995.204 Hz
 FIDRES 0.091480 Hz
 AQ 5.4657526 sec
 RG 101.6
 DW 83.400 usec
 DE 6.00 usec
 TE 294.3 K
 D1 1.0000000 sec
 TD0 1

CHANNEL f1
 NUCL1 1H
 P1 14.00 usec
 PL1 0.00 dB
 SFO1 300.1322510 MHz
 F2 - Processing parameters
 SI 32768
 SF 300.130032 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

NMR300
sample name:wkb090322-No3-py-ol-hex
solvent:cdcl3

```

NAME      wkb090322-No3-py-c
EXPNO     2
PROCNO    1
F2Acq     20090324
Time      0.28
INSTRUM   5 mm BBD
PULPROG   zgpg30
TD         32768
SOLVENT   DMSO
NS         235
DS         0
SMH        18115.941 Hz
FIDRES     0.552855 Hz
AQ         0.9084468 sec
RG         161.3
IN         27.600 usec
TE         294.5 K
D1         1.00000000 sec
d11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         9.90 usec
PL1        -1.10 dB
SFO1       75.4756731 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        0.00 dB
PL12       15.80 dB
SFO2       300.1312005 MHz
SI         32768
SF         75.467525 MHz
WDM        EM
SSB        0
LB         1.00 Hz
GB         0
PC         0.10
  
```

160.634
147.889
136.692
134.054
131.357
122.256
120.890
77.423
77.000
76.576
73.953
34.249
22.144
13.676

sample: wxb-090324-No1-up
 solvent: CDCl₃

Bruker 300M szpku
 sample name: wxb090219-Sketone-up
 solvent: cdcl3

NMR300
 sample name: wxb090219-Sketone-up
 solvent: cdcl3

164.103
 164.051
 160.635
 160.785

145.692
 145.529

136.765
 128.924
 128.814
 128.705
 126.475
 125.964
 125.863
 125.818

115.552
 115.267

77.424
 77.000
 76.577
 75.779
 75.707

Bruker 300M szpkv
 sample name: wxb090402-No4-thia-pyro-DMA-amide
 solvent: cdc13

```

NAME      wxb090402-No4-thia-pyro-DMA-?
EXPNO     1
PROCNO    1
Date_     20090403
Time      18.58
INSTRUM   spect
PROBHD    5 mm PABUL 13C
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         0
SWH        5995.204 Hz
FIDRES     0.091480 Hz
AQ         5.4657526 sec
RG         161.3
DE         83.400 usec
TE         293.2 K
D1         1.00000000 sec
TDO        1

===== CHANNEL f1 =====
NUC1       1H
P1         14.00 usec
PL1        0.00 dB
SFO1       300.132510 MHz
SI         32768
SF         300.130043 MHz
WDW         EM
SSB         0
GB         0
PC         1.00
  
```

NMR300
 sample name: wbd090402-No4-Chia-pyro-DMA-amide
 solvent: cdcl3

NAME wbd090402-No4-Chia-pyro-DMA-amide
 EXPTNO 1
 PROCNO 2
 Date_ 20090403
 Time 19.00
 INSTRUM spect
 PROBRD 5 mm PNUCL 13C
 PULPROG zgpg30
 TD 32768
 SOLVENT DMSO
 NS 1783
 DS 0
 SFR 1815.944 Hz
 ZFDRS 0.55235 Hz
 AQ 0.90468 sec
 RG 4813
 FID 27.601 usec
 DE 6.00 usec
 TE 293.3 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 TDO 1

===== CHANNEL F1 =====
 NUCL 13C
 P1 9.90 usec
 PL1 -1.10 dB
 SFO1 75.4756731 MHz

===== CHANNEL F2 =====
 CPDPRG2 waltz16
 NUCL2 1H
 P2 80.00 usec
 PL2 0.00 dB
 SFO2 300.1212005 MHz
 ST 32768
 ST 75.467534 MHz
 ST 0
 SSB 0
 TBR 1.00 Hz
 GB 0
 PC 1.40

Bruker 300M szpku
 sample name: wxb0 90225-pyamide-up
 solvent: cdcl3

NMR300
 sample name: wxb090225-pyamide-up
 solvent: cdcl3