Supporting Information

Rhodium-catalyzed Intramolecular Alkyne-Carbodiimide Pauson-Khand Type Reaction

Takao Saito*, Katsuya Sugizaki, Takashi Otani, and Toshiyuki Suyama

*Department of Chemistry, Faculty of Science, Tokyo University of Science,
Kagurazaka, Shinjuku-ku, Tokyo 162-8601 Japan*

Table of Contents

General	S2
Spectral data of alkyne-carbodiimide substrates	S2
A typical procedure for the Pauson-Khand type reaction	S6
Spectral data of Pauson-Khand products	S6
¹ H- and ¹³ C-NMR spectra of alkyne-carbodiimide substrates	S13
¹ H- and ¹³ C-NMR spectra of Pauson-Khand products	S43

Experimental

General

All Melting points were determined on a Yanaco melting point apparatus and are uncorrected. Infrared spectra were recorded on Hitachi Model 270-30 or Horiba FT-710 model spectrophotometers. ^1H and ^{13}C NMR spectral data were obtained with Bruker Avance-600, JEOL JNM-EX 500, JEOL JNM-LA 400, JEOL JNM-EX 300, and/or JEOL JNM-EX 270 instruments and chemical shifts are reported in ppm down field from tetramethylsilane (TMS) using an internal standard of TMS and/or CDCl_3 (In other noticed/acetone d_6 in case of needed.). Mass spectra were measured on a Hitachi double-focusing spectrometer M-80B model spectrometer. Elemental analyses were performed with a YANACO CHN-CODER MT-6 model analyzer.

Carbodiimides **1a-j** and **3a-g** were prepared by the aza-Wittig reaction between corresponding iminophosphoranes and isocyanates as described in a previous paper.¹ Analytical data for the carbodiimides **1c** and **1h** have been reported.²

(2-Phenylethynylphenyl)propylcarbodiimide (**1a**)

Colorless oil.

IR (neat/ cm^{-1}): 3062, 2252, 2144, 1164, 910, 647.

^1H -NMR (500 MHz, CDCl_3): δ 0.90 (t, $J = 7.2$ Hz, 3H), 1.61 (tt, $J = 6.9, 7.2$ Hz, 2H), 3.30 (t, $J = 6.9$ Hz, 2H), 7.04-7.08 (m, 2H), 7.21-7.25 (m, 1H), 7.31-7.35 (m, 3H), 7.46-7.48 (m, 1H), 7.58-7.60 (m, 2H).

^{13}C -NMR (125.7 MHz, CDCl_3): δ 11.29 (CH_3), 24.39 (CH_2), 48.42 (CH_2), 86.34 (C), 95.12 (C), 118.73 (C), 123.21 (C), 123.95 (CH), 124.08 (CH), 128.23 (CH), 128.35 (CH), 129.23 (CH), 131.48 (CH), 133.15 (CH), 133.42 (C), 141.37 (C).

HRMS-ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{18}\text{H}_{16}\text{N}_2\text{Na}$: 283.1206, found: 283.1200.

Cyclohexyl(2-phenylethynylphenyl)carbodiimide (**1b**)

Colorless oil.

IR (neat/ cm^{-1}): 3062, 2129, 2252, 1164, 910, 647.

^1H -NMR (500 MHz, CDCl_3): δ 1.11-1.24 (m, 3H), 1.36-1.54 (m, 3H), 1.61-1.74 (m, 2H), 1.85-1.98 (m, 2H), 3.43-3.46 (m, 1H), 7.05 (ddd, $J = 1.2, 7.5, 7.5$ Hz, 1H), 7.09 (dd, $J = 1.0, 8.1$ Hz, 1H), 7.23 (dd, $J = 1.5, 7.7$ Hz, 1H), 7.29-7.37 (m, 3H), 7.47 (dd, $J = 1.5, 7.7$ Hz, 1H), 7.55-7.65 (m, 2H).

^{13}C -NMR (125.7 MHz, CDCl_3): δ 24.22 (CH_2), 25.20 (CH_2), 34.51 (CH_2), 56.43 (CH), 86.43 (C), 94.99 (C), 118.58 (C), 123.28 (C), 123.87 (CH), 124.03 (CH), 128.20 (CH), 128.31 (CH), 129.19 (CH), 131.58 (CH), 133.16 (CH), 133.41 (C), 141.59 (C).

HRMS-ESI (m/z): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{21}\text{H}_{20}\text{N}_2\text{Na}$: 323.1519, found: 323.1520.

¹ Saito, T.; Shiotani, M.; Otani, T.; Hasaba, S. *Heterocycles* **2003**, 60, 1045.

² Shi, C.; Zhang, Q.; Wang, K.K. *J. Org. Chem.* **1999**, 64, 925.

(2-Phenylethynylphenyl)-p-tolylcarbodiimide (**1d**)

Colorless oil.

IR (neat/cm⁻¹): 3062, 2252, 2136, 1157, 910, 647.

¹H-NMR (600 MHz, CDCl₃): δ 2.25 (s, 3H), 6.98 (d, *J* = 8.0 Hz, 2H), 7.04 (d, *J* = 8.0 Hz, 2H), 7.10-7.15 (m, 2H), 7.16-7.21 (m, 2H), 7.21-7.28 (m, 2H), 7.32 (d, *J* = 7.2 Hz, 2H), 7.50 (d, *J* = 7.7 Hz, 1H).

¹³C-NMR (150.9 MHz, CDCl₃): δ 20.86 (CH₃), 85.63 (C), 96.47 (C), 119.82 (C), 122.74 (C), 124.11 (CH), 124.52 (CH), 125.11 (CH), 127.97 (CH), 128.24 (CH), 129.24 (CH), 129.88 (CH), 131.56 (CH), 133.19 (C), 133.25 (CH), 134.94 (C), 135.71 (C), 139.17 (C).

HRMS-ESI (*m/z*): [M+Na]⁺ calcd for C₂₂H₁₆N₂Na: 331.1206, found: 331.1216.

(4-Chloro-phenyl)(2-phenylethynylphenyl)carbodiimide (**1e**)

Colorless solid, mp 73-75 °C.

IR (KBr/cm⁻¹): 2252, 2136, 1095, 910.

¹H-NMR (500 MHz, CDCl₃): δ 7.04-7.05 (m, 2H), 7.09-7.23 (m, 6H), 7.25-7.32 (m, 4H), 7.52 (dd, *J* = 1.5, 7.9 Hz, 1H).

¹³C-NMR (125.7 MHz, CDCl₃): δ 85.35 (C), 96.79 (C), 120.16 (C), 122.50 (C), 124.57 (CH), 125.43 (CH), 125.52 (CH), 128.07 (CH), 128.49 (CH x 2), 129.32 (CH), 130.47 (C), 131.44 (CH), 132.88 (C), 133.22 (CH), 137.43 (C), 138.34 (C).

HRMS-ESI (*m/z*): [M+Na]⁺ calcd for C₂₁H₁₃ClN₂Na: 351.0659, found: 351.0659.

Propyl(2-trimethylsilanylethynylphenyl)carbodiimide (**1f**)

Colorless oil.

IR (neat/cm⁻¹): 3062, 2144, 1951, 1419, 1342, 1164, 856, 640.

¹H-NMR (500 MHz, CDCl₃): δ 0.27 (s, 9H), 1.01 (t, *J* = 7.3 Hz, 3H), 1.71 (tt, *J* = 6.7, 7.3 Hz, 2H), 3.40 (t, *J* = 7.5 Hz, 2H), 7.00-7.05 (m, 2H), 7.22 (dt, *J* = 1.6, 7.5 Hz, 1H), 7.42 (d, *J* = 1.4, 7.5 Hz, 1H).

¹³C-NMR (125.7 MHz, CDCl₃): δ 0.01 (CH₃), 11.44 (CH₃), 24.49 (CH₂), 48.47 (CH₂), 100.20 (C), 101.94 (C), 118.33 (C), 123.85 (CH), 123.95 (CH), 129.42 (CH), 133.41 (C), 133.89 (CH), 141.78 (C).

HRMS-ESI (*m/z*): [M+H]⁺ calcd for C₁₅H₂₁N₂Si: 257.1469, found: 257.1460.

Cyclohexyl(2-trimethylsilanylethynylphenyl)carbodiimide (**1g**)

Colorless oil.

IR (neat/cm⁻¹): 3062, 2360, 2136, 1419, 1357, 1164, 863, 640.

¹H-NMR (500 MHz, CDCl₃): δ 0.27 (s, 9H), 1.20-1.39 (m, 3H), 1.42-1.59 (m, 3H), 1.68-1.80 (m, 2H), 1.91-2.06 (m, 2H), 3.52 (dddd, J = 3.6, 3.6, 9.6, 9.6 Hz, 1H), 7.00 (dd, J = 7.7, 7.7 Hz, 1H), 7.06 (d, J = 7.7 Hz, 1H), 7.21 (dd, J = 1.5, 7.7 Hz, 1H), 7.41 (dd, J = 7.7, 7.7 Hz, 1H).

¹³C-NMR (125.7 MHz, CDCl₃): δ -0.03 (CH₃), 24.23 (CH₂), 25.30 (CH₂), 34.61 (CH₂), 56.34 (CH), 99.93 (C), 101.92 (C), 118.13 (C), 123.72 (CH), 123.86 (CH), 129.34 (CH), 133.32 (C), 133.79 (CH), 141.97 (C).

HRMS-ESI (m/z): [M+Na]⁺ calcd for C₁₈H₂₄N₂NaSi: 319.1601, found: 319.1590.

p-Tolyl-(2-trimethylsilanylethynylphenyl)carbodiimide (**1i**)

Colorless oil.

IR (neat/cm⁻¹): 2962, 2360, 2252, 1488, 1249, 1218, 910, 732.

¹H-NMR (500 MHz, CDCl₃): δ 0.12 (s, 9H), 2.35 (s, 3H), 7.09-7.15 (m, 6H), 7.24 (ddd, J = 1.5, 7.6, 7.6 Hz, 1H), 7.48 (dd, J = 1.5, 7.6 Hz, 1H).

¹³C-NMR (125.7 MHz, CDCl₃): δ -0.44 (CH₃), 20.93 (CH₃), 100.81 (C), 102.24 (C), 119.59 (C), 123.89 (CH), 124.22 (CH), 124.55 (CH), 125.00 (CH), 129.45 (CH), 133.01 (C), 133.85 (CH), 135.03 (C), 136.05 (C), 139.33 (C).

HRMS-ESI (m/z): [M+Na]⁺ calcd for C₁₉H₂₀N₂NaSi: 327.1288, found: 327.1277.

(4-Chloro-phenyl)(2-trimethylsilanylethynylphenyl)carbodiimide (**1j**)

Colorless oil.

IR (neat/cm⁻¹): 3070, 2252, 2144, 1142, 1380, 1149, 863, 647.

¹H-NMR (300 MHz, CDCl₃): δ 0.03 (s, 9H), 6.99-7.09 (m, 4H), 7.15-7.24 (m, 3H), 7.35-7.44 (m, 1H).

¹³C-NMR (75.5 MHz, CDCl₃): δ -0.47 (CH₃), 100.57 (C), 102.71 (C), 119.94 (C), 124.63 (CH), 125.43 (CH), 125.53 (CH), 129.48 (CH), 129.50 (CH), 130.47 (C), 132.43 (C), 133.85 (CH), 137.92 (C), 138.25 (C).

HRMS-ESI (m/z): [M+Na]⁺ calcd for C₁₈H₁₇ClN₂NaSi: 347.0742, found: 347.0744.

Pent-3-ynylpropylcarbodiimide (**3a**)

Colorless oil.

IR (neat, cm⁻¹): 2252, 2159.

¹H-NMR (600 MHz, CDCl₃): δ 0.96 (t, J = 7.4 Hz, 3H), 1.62 (tq, J = 7.3, 7.3 Hz, 2H), 1.80 (t, J = 2.6 Hz, 3H), 2.39 (m, 2H), 3.20 (t, J = 6.9 Hz, 2H), 3.29 (t, J = 6.8 Hz, 2H).

¹³C-NMR (75.5 MHz, CDCl₃): δ 3.39 (CH₃), 11.30 (CH₃), 21.52 (CH₂), 24.40 (CH₂), 45.90 (CH₂), 48.47 (CH₂), 76.06 (C), 77.25 (C), 140.23 (C).

Pent-3-ynylphenylcarbodiimide (**3b**)

Colorless oil.

IR (neat, cm^{-1}): 3154, 2252, 2136, 1157, 910.

$^1\text{H-NMR}$ (300 MHz, CDCl_3): δ 1.63 (t, $J = 2.5$ Hz, 3H), 2.48-2.55 (m, 2H), 3.47 (t, $J = 6.3$ Hz, 2H), 7.07-7.17 (m, 3H), 7.25-7.32 (m, 2H).

$^{13}\text{C-NMR}$ (75.5 MHz, CDCl_3): δ 3.37 (CH_3), 21.69 (CH_2), 46.12 (CH_2), 75.57 (C), 78.39 (C), 123.79 (CH), 124.68 (CH), 129.20 (CH), 136.93 (C), 140.23 (C).

HRMS-ESI (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{12}\text{H}_{13}\text{N}_2$: 185.1073, found: 185.1079.

Pent-3-ynyl-p-tolylcarbodiimide (**3c**)

Colorless oil.

IR (neat/ cm^{-1}): 3154, 2252, 2129, 1157, 910, 647.

$^1\text{H-NMR}$ (600 MHz, CDCl_3): δ 1.66 (t, $J = 2.4$ Hz, 3H), 2.31 (s, 3H), 2.48-2.52 (m, 2H), 3.46 (t, $J = 6.3$ Hz, 2H), 7.04 (d, $J = 8.3$ Hz, 2H), 7.08 (d, $J = 8.3$ Hz, 2H).

$^{13}\text{C-NMR}$ (150.9 MHz, CDCl_3): δ 3.40 (CH_3), 20.90 (CH_3), 21.72 (CH_2), 46.19 (CH_2), 75.67 (C), 78.29 (C), 123.58 (CH), 129.81 (CH), 134.41 (C), 137.35 (C), 137.40 (C).

HRMS-ESI (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{13}\text{H}_{15}\text{N}_2$: 199.1230, found: 199.1238.

(4-Chlorophenyl)pent-3-ynylcarbodiimide (**3d**)

Colorless oil.

IR (neat/ cm^{-1}): 3154, 2252, 2136, 1157, 910, 647.

$^1\text{H-NMR}$ (600 MHz, CDCl_3): δ 1.64 (t, $J = 2.6$ Hz, 3H), 2.49-2.53 (m, 2H), 3.47 (t, $J = 6.2$ Hz, 2H), 7.08 (d, $J = 8.8$ Hz, 2H), 7.24 (d, $J = 8.8$ Hz, 2H).

$^{13}\text{C-NMR}$ (150.9 MHz, CDCl_3): δ 3.41 (CH_3), 21.70 (CH_2), 46.08 (CH_2), 75.51 (C), 78.52 (C), 124.99 (CH), 129.25 (CH), 129.88 (C), 136.30 (C), 139.12 (C).

HRMS-ESI (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{12}\text{H}_{12}\text{ClN}_2$: 219.0684, found: 219.0684.

Hex-3-ynylphenylcarbodiimide (**3e**)

Colorless oil.

IR (neat/ cm^{-1}): 3062, 2136, 1157, 902, 601.

$^1\text{H-NMR}$ (600 MHz, CDCl_3): δ 1.00 (t, $J = 7.6$ Hz, 3H), 2.02 (tq, $J = 2.5, 7.6$ Hz, 2H), 2.52 (tt, $J = 2.5, 6.5$ Hz, 2H), 3.46 (t, $J = 6.5$ Hz, 2H), 7.08-7.11 (m, 1H), 7.13-7.15 (m, 2H), 7.26-7.29 (m, 2H).

$^{13}\text{C-NMR}$ (150 MHz, CDCl_3): 12.27 (CH_2), 13.81 (CH_3), 21.70 (CH_2), 46.08 (CH_2), 75.67 (C), 84.30 (C), 123.72 (CH), 124.60 (CH), 129.13 (CH), 136.68 (C), 140.27 (C).

HRMS-ESI (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{13}\text{H}_{15}\text{N}_2$: 199.1230, found: 199.1222.

Hex-3-ynyl-p-tolylcarbodiimide (**3f**)

Colorless oil.

IR (neat/ cm^{-1}): 3154, 2252, 2129, 1157, 910, 647.

$^1\text{H-NMR}$ (600 MHz, CDCl_3): δ 1.02 (t, $J = 7.6$ Hz, 3H), 2.04 (tq, $J = 2.6, 7.6$ Hz, 2H), 2.31 (s, 3H), 2.52 (tt, $J = 2.6, 6.4$ Hz, 2H), 3.46 (t, $J = 6.4$ Hz, 2H), 7.03 (d, $J = 8.3$ Hz, 2H), 7.07 (d, $J = 8.3$ Hz, 2H).

$^{13}\text{C-NMR}$ (150 MHz, CDCl_3): δ 12.33 (CH_2), 13.88 (CH_3), 20.86 (CH_3), 21.74 (CH_2), 46.18 (CH_2), 75.79 (C), 84.24 (C), 123.52 (CH), 129.76 (CH), 134.35 (C), 137.22 (C), 137.36 (C).

HRMS-ESI (m/z): $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{14}\text{H}_{17}\text{N}_2$: 213.1386, found: 213.1376.

(4-Chlorophenyl)hex-3-ynylcarbodiimide (**3g**)

Colorless oil.

IR (neat/cm⁻¹): 3016, 2399, 2136, 1157, 910, 671.

¹H-NMR (600 MHz, CDCl₃): δ 1.01 (t, J = 7.4 Hz, 3H), 2.03 (tq, J = 2.5, 7.4 Hz, 2H), 2.53 (tt, J = 2.5, 6.2 Hz, 2H), 3.47 (t, J = 6.2 Hz, 2H), 7.07 (d, J = 8.9 Hz, 2H), 7.23 (d, J = 8.9 Hz, 2H).

¹³C-NMR (150 MHz, CDCl₃): δ 12.34 (CH₂), 13.86 (CH₃), 21.73 (CH₂), 46.08 (CH₂), 75.63 (C), 84.47 (C), 124.95 (CH), 129.22 (CH), 129.84 (C), 136.10 (C), 139.14 (C).

HRMS-ESI (m/z): [M+H]⁺ calcd for C₁₃H₁₄ClN₂: 233.0840, found: 233.0838.

A typical procedure for the Pauson-Khand type reaction of 1a

A flask containing a mixture of [RhCl(cod)]₂ (5.60 mg, 0.011 mmol) and 1,3-bis(diphenylphosphino)propane (dppp) (10.7 mg, 0.026 mmol) was exhausted and back-filled with carbon monoxide (three-times). Toluene (0.4 mL) was added and the mixture was stirred at room temperature for 30 min under an atmosphere of carbon monoxide. After addition of a toluene solution (1.0 mL) of carbodiimide **1a** (61.7 mg, 0.237 mmol), the reaction mixture was heated at 120 °C (bath temperature) for 1.5 h. Evaporation of the reaction mixture and column chromatography of the residue on silica gel with hexane-EtOAc (10:1) as an eluant gave **2a** (52.6 mg, 77% yield).

3-Phenyl-1-propylpyrrolo[2,3-b]indol-2-one (**2a**)

Brown crystals, mp 128-129 °C.

IR (KBr/cm⁻¹): 2904, 1724, 1626, 1586, 1426, 1346, 1300, 1154, 750.

¹H-NMR (400 MHz, CDCl₃): δ 0.99 (t, J = 7.3 Hz, 3H), 1.79 (sextet, J = 7.3 Hz, 2H), 3.62 (t, J = 7.3 Hz, 2H), 6.91 (ddd, J = 1.2, 1.2, 7.3 Hz, 1H), 7.14-7.22 (m, 1H), 7.20 (ddd, J = 1.2, 7.3, 7.3 Hz, 1H), 7.47-7.57 (m, 4H), 7.90-7.93 (m, 2H).

¹³C-NMR (100 MHz, CDCl₃): δ 11.27 (CH₃), 21.69 (CH₂), 41.70 (CH₂), 120.13 (CH), 124.60 (CH), 124.63 (C), 124.93 (CH), 128.88 (CH₂), 129.15 (C), 129.73 (C), 129.76 (CH₂), 130.55 (CH), 132.99 (CH), 138.20 (C), 161.92 (C), 172.02 (C), 173.33 (C).

LRMS-EI m/z (ion, % relative intensity): 288 (M⁺, 100%), 259 (M⁺-29, 65%), 246 (M⁺-42, 51%), 232 (M⁺-56, 78%), 218 (M⁺-70, 34%), 204 (M⁺-84, 38%).

HRMS-EI (m/z): [M]⁺ calcd for C₁₉H₁₆N₂O: 288.1263, found: 288.1247.

3-Phenyl-1-cyclohexylpyrrolo[2,3-b]indol-2-one (**2b**)

Brown crystals, mp 154-157 °C.

IR (KBr/cm⁻¹): 2912, 1718, 1620, 1576, 1414, 1296, 754, 690.

¹H-NMR (600 MHz, CDCl₃): δ 1.26-1.42 (m, 3H), 1.67-1.69 (m, 1H), 1.84-1.89 (m, 4H), 2.10-2.17 (m, 2H), 3.99 (tt, J = 3.7, 12.2 Hz, 1H), 6.90 (dt, J = 1.3, 7.3 Hz, 1H), 7.16-7.21 (m, 2H), 7.45-7.48 (m, 1H), 7.49-7.52 (m, 2H), 7.55 (d, J = 7.4 Hz, 1H), 7.88-7.90 (m, 2H).

¹³C-NMR (150 MHz, CDCl₃): δ 24.97 (CH₂), 25.78 (CH₂), 30.30 (CH₂), 52.10 (CH), 120.10 (CH), 124.47 (CH), 124.52 (C), 124.71 (CH), 128.76 (C), 128.82 (CH₂), 129.75 (CH₂), 129.77 (C), 130.40 (CH), 132.89 (CH), 138.73 (C), 162.29 (C), 171.64 (C), 173.02 (C).

LRMS-EI m/z (ion, % relative intensity): 328 (M⁺, 100%), 299 (M⁺-29, 29%), 274 (M⁺-54, 9%), 247

($M^+ - 81$, 45%), 218 ($M^+ - 110$, 35%), 190 ($M^+ - 138$, 12%).
HRMS-EI (m/z): [M] $^+$ calcd for $C_{22}H_{20}N_2O$: 328.1576, found: 328.1585.

1,3-Diphenylpyrrolo[2,3-*b*]indol-2-one (2c**)**

Brown crystals, mp 118-120 °C.

IR (KBr/ cm^{-1}): 3060, 1726, 1620, 1578, 1488, 1414, 1304, 738, 682.

^1H -NMR (500 MHz, $CDCl_3$): δ 6.93-6.98 (m, 1H), 7.23-7.25 (m, 2H), 7.29-7.33 (m, 1H), 7.46-7.55 (m, 5H), 7.63 (dd, $J = 1.5, 7.3$ Hz, 1H), 7.73 (dd, $J = 1.2, 7.3$ Hz, 2H), 7.95 (dd, $J = 1.5, 6.1$ Hz, 2H).

^{13}C -NMR (125 MHz, $CDCl_3$): δ 120.70 (CH), 123.68 (CH $_x$ 2), 124.24(C), 124.98 (CH), 125.01 (CH), 126.79 (CH), 128.47 (C), 128.96 (CH $_x$ 2), 129.10 (CH $_x$ 2), 129.21 (C), 129.90 (CH $_x$ 2), 130.80 (CH), 133.31 (CH), 133.49 (C), 138.68 (C), 161.36 (C), 170.68 (C), 171.86 (C).

LRMS-EI m/z (ion, % relative intensity): 322 (M^+ , 100%), 293 ($M^+ - 29$, 75%), 147 ($M^+ - 146$, 17%), 28 ($M^+ - 294$, 65%).

HRMS-EI (m/z): [M] $^+$ calcd for $C_{22}H_{14}N_2O$: 322.1106, found: 322.1103.

3-Phenyl-1-*p*-tolylpyrrolo[2,3-*b*]indol-2-one (2d**)**

Brown crystals, mp 206-208 °C.

IR (KBr/ cm^{-1}): 3050, 1720, 1576, 1420, 758.

^1H -NMR (600 MHz, $CDCl_3$): δ 2.38 (s, 3H), 6.97 (dt, $J = 2.0, 7.2$ Hz, 1H), 7.22-7.25 (m, 2H), 7.29 (d, $J = 8.0$ Hz, 2H), 7.49-7.59 (m, 5H), 7.64 (d, $J = 7.6$ Hz, 1H), 7.97 (dd, $J = 1.5, 8.3$ Hz, 2H).

^{13}C -NMR (150 MHz, $CDCl_3$): δ 21.11 (CH $_3$), 120.66 (CH), 123.80 (CH $_x$ 2), 124.34(C), 124.90 (CH), 125.00 (CH), 128.95 (CH $_x$ 2), 129.52 (C), 129.82 (CH $_x$ 2), 129.90 (CH $_x$ 2), 130.75 (CH), 130.79(C), 133.27 (CH), 136.80 (C), 138.674 (C), 161.51 (C), 170.93 (C), 171.98 (C). (one signal is missing)

LRMS-EI m/z (ion, % relative intensity): 336 (M^+ , 100%), 307 ($M^+ - 29$, 55%), 146 ($M^+ - 190$, 9%).

HRMS-EI (m/z): [M] $^+$ calcd for $C_{23}H_{16}N_2O$: 336.1263, found: 336.1255.

1-(*p*-Chlorophenyl)-3-phenylpyrrolo[2,3-*b*]indol-2-one (2e**)**

Brown solid. mp 227-230 °C.

IR (KBr/ cm^{-1}): 3028, 1718, 1570, 1486, 1410, 1086, 754.

^1H -NMR (600 MHz, $CDCl_3$): δ 6.98 (dt, $J = 1.3, 7.3$ Hz, 1H), 7.23-7.28 (m, 2H), 7.45 (td, $J = 2.5, 8.7$ Hz, 2H), 7.50-7.57 (m, 3H), 7.64 (d, $J = 7.2$ Hz, 1H), 7.76 (td, $J = 2.5, 8.7$ Hz, 2H), 7.95 (dd, $J = 1.3, 7.3$

Hz, 2H).

^{13}C -NMR (150 MHz, CDCl_3): δ 120.78 (CH), 124.11 (C), 124.48 (CH_2), 125.10 (CH), 125.15 (CH), 128.99 (CH_2), 129.25 (C), 129.29 (CH_2), 129.87 (CH_2), 130.92 (CH), 132.05 (C), 132.24 (C), 133.42 (CH), 138.73 (C), 161.18 (C), 170.22 (C), 171.65 (C). (One signal overlapped)

LRMS-EI m/z (ion, % relative intensity): 356 (M^+ , 100%), 327 ($\text{M}^+ - 29$, 14%), 293 ($\text{M}^+ - 63$, 84%), 190 ($\text{M}^+ - 166$, 12%), 164 ($\text{M}^+ - 192$, 10%).

HRMS-EI (m/z): $[\text{M}]^+$ calcd for $\text{C}_{22}\text{H}_{13}\text{N}_2\text{ClO}$: 356.0716, found: 356.0709.

1-Propyl-3-(trimethylsilyl)pyrrolo[2,3-*b*]indol-2-one (2f)

Brown oil.

^1H -NMR (270 MHz, CDCl_3): δ 0.38 (s, 9H), 0.95 (t, $J = 7.3$ Hz, 3H), 1.73 (sextet, $J = 7.3$ Hz, 2H), 3.51 (t, $J = 7.3$ Hz, 2H), 6.88 (dt, $J = 0.8, 7.3$ Hz, 1H), 7.08 (d, $J = 7.1$ Hz, 1H), 7.18 (dt, $J = 1.0, 7.6$ Hz, 1H), 7.37 (dd, $J = 0.5, 7.3$ Hz, 1H).

^{13}C -NMR (67 MHz, CDCl_3): δ -0.97 (CH_3), 11.20 (CH_3), 21.61 (CH_2), 41.35 (CH_2), 119.77 (CH), 124.30 (CH), 125.18 (C), 126.02 (CH), 133.19 (CH), 133.55 (C), 154.59 (C), 162.52 (C), 172.07 (C), 177.38 (C).

LRMS-EI m/z (ion, % relative intensity): 284 (M^+ , 100%), 269 ($\text{M}^+ - 15$, 27%), 255 ($\text{M}^+ - 29$, 20%), 228 ($\text{M}^+ - 56$, 74%), 199 ($\text{M}^+ - 85$, 35%), 183 ($\text{M}^+ - 101$, 20%).

HRMS-EI (m/z): $[\text{M}]^+$ calcd for $\text{C}_{16}\text{H}_{20}\text{N}_2\text{OSi}$: 284.1345, found: 284.1350.

1-Cyclohexyl-3-(trimethylsilyl)pyrrolo[2,3-*b*]indol-2-one (2g)

Brown oil.

^1H -NMR (600 MHz, CDCl_3): δ 0.37 (s, 9H), 1.23-1.37 (m, 3H), 1.64-1.66 (m, 1H), 1.77-1.79 (m, 2H), 1.83-1.85 (m, 2H), 2.02 (dd, $J = 3.5, 12.3$ Hz, 1H), 2.07 (dd, $J = 3.5, 12.3$ Hz, 1H), 3.89 (tt, $J = 3.9, 12.3$ Hz, 1H), 6.86 (dt, $J = 1.2, 7.5$ Hz, 1H), 7.09 (d, $J = 7.6$ Hz, 1H), 7.15 (dt, $J = 1.2, 7.6$ Hz, 1H), 7.34 (d, $J = 7.6$ Hz, 1H).

^{13}C -NMR (150 MHz, CDCl_3): δ -0.90 (CH_3), 24.95 (CH_2), 25.76 ($\text{CH}_2 \times 2$), 30.26 ($\text{CH}_2 \times 2$), 51.66 (CH), 119.81 (CH), 124.22 (CH), 125.14 (C), 125.89 (CH), 133.02 (C), 133.13 (CH), 155.08 (C), 162.99 (C), 171.74 (C), 177.26 (C).

LRMS-EI m/z (ion, % relative intensity): 324 (M^+ , 100%), 309 ($\text{M}^+ - 15$, 12%), 280 ($\text{M}^+ - 44$, 12%), 227 ($\text{M}^+ - 97$, 36%), 199 ($\text{M}^+ - 125$, 29%).

HRMS-EI (m/z): $[\text{M}]^+$ calcd for $\text{C}_{19}\text{H}_{24}\text{N}_2\text{OSi}$: 324.1658, found: 324.1659.

1-Phenyl-3-(trimethylsilyl)pyrrolo[2,3-*b*]indol-2-one (**2h**)

Brown crystals, mp 109-112 °C

IR (KBr/cm⁻¹): 2916, 1724, 1586, 1488, 1398, 1246, 744.

¹H-NMR (270 MHz, CDCl₃): δ 0.36 (s, 9H), 6.86 (dt, *J* = 1.2, 7.3 Hz, 1H), 7.05 (m, 3H), 7.37 (m, 3H), 7.60 (dd, *J* = 1.2, 8.7 Hz, 2H).

¹³C-NMR (150 MHz, acetone *d*₆): δ -1.09 (CH₃), 120.76 (CH), 123.74 (CHx2), 125.52 (CH), 125.54 (C), 126.71 (CH), 127.03 (C), 129.36 (CHx2), 134.12 (CH), 134.24 (C), 135.07 (C), 155.28 (C), 163.00 (C), 171.18 (C), 176.47 (C).

LRMS-EI *m/z* (ion, % relative intensity): 318 (M⁺, 100%), 303 (M⁺-15, 19%), 289 (M⁺-29, 51%), 275 (M⁺-43, 32%), 259 (M⁺-59, 9%), 245 (M⁺-73, 9%).

HRMS-EI (*m/z*): [M]⁺ calcd for C₁₉H₁₈N₂OSi: 318.1188, found: 318.1193.

1-*p*-Tolyl-3-(trimethylsilyl)pyrrolo[2,3-*b*]indol-2-one (**2i**)

Brown crystals, mp 106-109 °C.

IR (KBr/cm⁻¹): 2844, 1720, 1588, 1402, 1116, 816.

¹H-NMR (270 MHz, CDCl₃): δ 0.36 (s, 9H), 2.29 (s, 3H), 6.86 (dt, *J* = 1.2, 7.5, Hz 1H), 7.08 (d, *J* = 7.3, 1H), 7.13-7.19 (m, 3H), 7.38 (d, *J* = 7.3 Hz, 1H), 7.43-7.46 (m, 2H).

¹³C-NMR (150 MHz, acetone *d*₆): δ -1.02 (CH₃), 20.83 (CH₃), 120.74 (CH), 123.71 (CHx2), 125.46 (CH), 125.60 (C), 127.00 (CH), 129.86 (CHx2), 132.49 (C), 134.09 (CH), 134.14 (C), 136.40 (C), 155.25 (C), 163.11 (C), 171.30 (C), 176.52 (C).

LRMS-EI *m/z* (ion, % relative intensity): 332 (M⁺, 100%), 289 (M⁺-41, 32%), 259 (M⁺-73, 9%), 73 (M⁺-259, 12%).

HRMS-EI (*m/z*): [M]⁺ calcd for C₂₀H₂₀N₂OSi: 332.1345, found: 332.1349.

1-*p*-(Chlorophenyl)-3-(trimethylsilyl)pyrrolo[2,3-*b*]indol-2-one (**2j**)

Brown crystals, mp 133-136 °C

IR (KBr/cm⁻¹): 2884, 1718, 1586, 1486, 1402, 820, 720.

¹H-NMR (270 MHz, CDCl₃): δ 0.37 (s, 9H), 6.89 (dt, *J* = 1.2, 7.3 Hz, 1H), 7.10 (d, *J* = 7.3 Hz, 1H), 7.17 (dt, *J* = 1.2, 7.3 Hz, 1H), 7.32-7.36 (m, 2H), 7.39 (d, *J* = 7.3 Hz, 1H), 7.62-7.66 (m, 2H).

¹³C-NMR (150 MHz, acetone *d*₆): δ -1.12 (CH₃), 120.87 (CH), 124.78 (CHx2), 125.43 (CH), 125.69 (C), 127.12 (CH), 129.39 (C), 131.30 (CHx2), 134.04 (C), 134.21 (C), 134.38 (CH), 155.34 (C), 162.79 (C), 170.78 (C), 176.33 (C).

LRMS-EI *m/z* (ion, % relative intensity): 352 (M⁺, 100%), 317 (M⁺-35, 54%), 289 (M⁺-28, 19%), 216 (M⁺-136, 7%), 73 (M⁺-279, 19%).

HRMS-EI (*m/z*): [M]⁺ calcd for C₁₉H₁₇ClN₂OSi: 352.0799, found: 352.0807.

3-Methyl-1-phenyl-4,5-dihydro-1*H*-pyrrolo[2,3-*b*]pyrrol-2-one (**4b**)

Colorless crystal, mp 94-95 °C

IR (KBr/cm⁻¹): 1728, 1540.

¹H-NMR (270 MHz, CDCl₃): δ 2.03 (t, *J* = 2.2 Hz, 3H), 2.80 (ddq, *J* = 4.4, 4.4, 2.2 Hz, 2H), 4.36 (dd, *J* = 4.4, 4.4 Hz, 2H), 7.24-7.28 (m, 1H), 7.32-7.46 (m, 2H), 7.68-7.70 (m, 2H).

¹³C-NMR (67 MHz, CDCl₃): δ 9.57 (CH₃), 24.42 (CH₂), 62.85 (CH₂), 123.38 (CHx2), 123.95 (C), 126.15 (CH), 128.92 (CHx2), 134.55 (C), 145.79 (C), 166.79 (C), 175.02 (C)

LRMS-EI *m/z* (ion, % relative intensity): 212 (M⁺, 100%), 183 (M⁺-29, 23%), 169 (M⁺-43, 7%), 156 (M⁺-56, 9%), 77 (M⁺-135, 22%), 53 (M⁺-159, 20%)

HRMS-EI (*m/z*): [M]⁺ calcd for C₁₃H₁₂N₂O: 212.0950, found: 212.0956.

3-Methyl-1-*p*-tolyl-4,5-dihydro-1*H*-pyrrolo[2,3-*b*]pyrrol-2-one (**4c**)

Colorless crystal, mp 110-112 °C

IR (KBr/cm⁻¹): 1730, 1698, 1634.

¹H-NMR (270 MHz, CDCl₃): δ 2.01 (t, *J* = 2.4 Hz, 3H), 2.35 (s, 3H), 2.77 (ddq, *J* = 4.6, 4.6, 2.4 Hz, 2H), 4.33 (dd, *J* = 4.6, 4.6 Hz, 2H), 7.23 (d, *J* = 8.2 Hz, 2H), 7.54 (d, *J* = 8.2 Hz, 2H).

¹³C-NMR (67 MHz, CDCl₃): δ 9.55 (CH₃), 20.96 (CH₃), 24.38 (CH₂), 62.79 (CH₂), 123.38 (CHx2), 123.84 (C), 129.46 (CHx2), 131.86 (C), 135.92 (C), 145.56 (C), 166.92 (C), 174.89 (C).

LRMS-EI: *m/z* (ion, % relative intensity): 226 (M⁺, 100%), 211 (M⁺-15, 15%), 197 (M⁺-29, 16%), 183 (M⁺-43, 11%), 170 (M⁺-56, 7%), 91 (M⁺-135, 15%), 53 (M⁺-173, 18%)

HRMS-EI (*m/z*): [M]⁺ calcd for C₁₄H₁₄N₂O: 226.1106, found: 226.1098.

1-(*p*-Chlorophenyl)-3-methyl-4,5-dihydro-1*H*-pyrrolo[2,3-*b*]pyrrol-2-one (**4d**)

Colorless crystals, mp 114-116 °C

IR (KBr/cm⁻¹): 2912, 1726, 1694, 1638.

¹H-NMR (270 MHz, CDCl₃): δ 2.02 (t, *J* = 2.2 Hz, 3H), 2.80 (ddq, *J* = 4.4, 4.4, 2.2 Hz, 2H), 4.36 (dd, *J* = 4.4, 4.4 Hz, 2H), 7.39 (d, *J* = 8.8 Hz, 2H), 7.73 (d, *J* = 8.8 Hz, 2H).

¹³C-NMR (125 MHz, CDCl₃): δ 9.58 (CH₃), 24.45 (CH₂), 62.93 (CH₂), 124.07 (C), 124.20 (CHx2), 129.01 (CHx2), 131.35 (C), 133.34 (C), 146.06 (C), 166.48 (C), 174.80 (C).

LRMS-EI: *m/z* (ion, % relative intensity): 246 (M⁺, 100%), 211 (M⁺-35, 28%), 183 (M⁺-63, 15%), 111 (M⁺-135, 15%), 75 (M⁺-171, 12%), 53 (M⁺-193, 25%).

HRMS-EI (*m/z*): [M]⁺ calcd for C₁₃H₁₁ClN₂O: 246.0560, found: 246.0561.

3-Ethyl-1-phenyl-4,5-dihydro-1*H*-pyrrolo[2,3-*b*]pyrrol-2-one (**4e**)

Colorless crystal, mp 99 -101 °C.

¹H-NMR (270 MHz, CDCl₃): δ 1.25 (t, *J* = 7.6 Hz, 3H), 2.47 (tq, *J* = 2.4, 7.6 Hz, 2H), 2.86 (ddt, *J* = 4.4, 4.4, 2.4 Hz, 2H), 4.36 (dd, *J* = 4.4, 4.4 Hz, 2H), 7.24-7.27 (m, 1H), 7.41-7.45 (m, 2H), 7.70 (d, *J* = 7.6 Hz, 2H).

¹³C-NMR (125 MHz, CDCl₃): δ 11.47 (CH₃), 17.91 (CH₂), 24.81 (CH₂), 63.15 (CH₂), 123.40 (CHx2), 126.10 (CH), 128.90 (CHx2), 129.68 (C), 134.55 (C), 144.71 (C), 167.00 (C), 174.59 (C).

LRMS-EI *m/z* (ion, % relative intensity): 226 (M⁺, 100%), 211 (M⁺-15, 78%), 197 (M⁺-29, 19%), 183 (M⁺-43, 12%), 77 (M⁺-149, 25%).

HRMS-EI (*m/z*): [M]⁺ calcd for C₁₄H₁₄N₂O: 226.1106, found: 226.1103.

3-Ethyl-1-*p*-tolyl-4,5-dihydro-1*H*-pyrrolo[2,3-*b*]pyrrol-2-one (**4f**)

Colorless crystal, mp 112-114 °C.

¹H-NMR (600 MHz, CDCl₃): δ 1.25 (t, *J* = 7.6 Hz, 3H), 2.35 (s, 3H), 2.47 (tq, *J* = 2.4, 7.6 Hz, 2H), 2.84-2.86 (m, 2H), 4.35 (dd, *J* = 4.0, 4.6 Hz, 2H), 7.23 (d, *J* = 8.2 Hz, 2H), 7.54 (d, *J* = 8.2 Hz, 2H).

¹³C-NMR (150 MHz, CDCl₃): δ 11.54 (CH₃), 17.96 (CH₃), 21.03 (CH₂), 24.87 (CH₂), 63.17 (CH₂), 123.56 (CHx2), 129.44 (CHx2), 129.73 (C), 131.92 (C), 136.03 (C), 144.54 (C), 167.22 (C), 174.76 (C).

LRMS-EI *m/z* (ion, % relative intensity): 240 (M⁺, 100%), 225 (M⁺-15, 76%), 211 (M⁺-29, 12%), 197, (M⁺-43, 13%), 91 (M⁺-149, 16%), 65 (M⁺-175, 12%).

HRMS-EI (*m/z*): [M]⁺ calcd for C₁₅H₁₆N₂O: 260.1263, found: 240.1267.

3-Ethyl-1-(*p*-chlorophenyl)-4,5-dihydro-1*H*-pyrrolo[2,3-*b*]pyrrol-2-one (**4g**)

Colorless crystal, mp 117-119 °C.

¹H-NMR (300 MHz, CDCl₃): δ 1.25 (t, *J* = 7.5 Hz, 3H), 2.46 (tq, *J* = 2.2, 7.5 Hz, 2H), 2.86 (ddt, *J* = 4.4, 4.4, 2.4 Hz, 2H), 4.36 (dd, *J* = 4.4, 4.4 Hz, 2H), 7.38 (d, *J* = 8.8 Hz, 2H), 7.72 (d, *J* = 8.8 Hz, 2H).

¹³C-NMR (75 MHz, CDCl₃): δ 11.44 (CH₃), 17.90 (CH₂), 24.81 (CH₂), 63.20 (CH₂), 124.21 (CHx2),

128.95 (CH_x2), 129.75 (C), 131.92 (C), 133.27 (C), 144.98 (C), 166.65 (C), 174.38 (C).
LRMS-EI: *m/z* (ion, % relative intensity): 260 (M⁺, 100%), 245 (M⁺-15, 63%), 225 (M⁺-35, 22%), 197 (M⁺-63, 10%).
HRMS-EI (*m/z*): [M]⁺ calcd for C₁₄H₁₃ClN₂O: 260.0716, found: 260.0725.

ObsNuc
ObsFreq
Solvent
Reference

¹³C
125.65 MHz
CDCl₃
77.0 ppm

1a

ObsNuc
ObsFreq
Solvent
Reference

¹H
500.0 MHz
CDCl₃
0.0 ppm

1b

ObsNuc
ObsFreq
Solvent
Reference

¹³C
125.65 MHz
CDCl₃
77.0 ppm

1b

ObsNuc
ObsFreq
Solvent
Reference

¹H
600.13 MHz
CDCl₃
0.0 ppm

1d

File E:\FOROL\1D\1D_13CD.RM1
Date 16/Feb/2007(Fri) 11:34:42
ObsNuc ¹³C
ObsFreq 150.9 MHz
ObsSet 0.0 kHz

1d

ObsNuc
ObsFreq
Solvent
Reference

^1H
500.0 MHz
 CDCl_3
0.0 ppm

1e

ObsNuc
ObsFreq
Solvent
Reference

¹³C
125.65 MHz
CDCl₃
77.0 ppm

1e

ObsNuc
ObsFreq
Solvent
Reference

¹H
500.0 MHz
CDCl₃
0.0 ppm

ObsNuc
ObsFreq
Solvent
Reference

¹³C
125.65 MHz
CDCl₃
77.0 ppm

ObsNuc
ObsFreq
Solvent
Reference

^1H
500.0 MHz
 CDCl_3
0.0 ppm

1g

ObsNuc	¹³ C
ObsFreq	125.65 MHz
Solvent	CDCl ₃
Reference	77.0 ppm

ObsNuc
ObsFreq
Solvent
Reference

¹H
500.0 MHz
CDCl₃
0.0 ppm

ObsNuc
ObsFreq
Solvent
Reference

¹³C
125.65 MHz
CDCl₃
77.0 ppm

ObsNuc
ObsFreq
Solvent
Reference

¹H
300.4 MHz
CDCl₃
0.0 ppm

ObsNuc
ObsFreq
Solvent
Reference

¹³C
75.45 MHz
CDCl₃
77.0 ppm

ObsNuc
ObsFreq
Solvent
Reference

¹H
600.13 MHz
CDCl₃
0.0 ppm

3a

ObsNuc
ObsFreq
Solvent
Reference

¹³C
150.9 MHz
CDCl₃
77.0 ppm

Hexane

3a

ObsNuc
ObsFreq
Solvent
Reference

¹H
300.4 MHz
CDCl₃
0.0 ppm

3b

ObsNuc
ObsFreq
Solvent
Reference

¹³C
75.45 MHz
CDCl₃
77.0 ppm

3b

ObsNuc
ObsFreq
Solvent
Reference

¹H
600.13 MHz
CDCl₃
0.0 ppm

3c

ObsNuc
ObsFreq
Solvent
Reference

¹³C
150.9 MHz
CDCl₃
77.0 ppm

3c

ObsNuc
ObsFreq
Solvent
Reference

¹H
600.13 MHz
CDCl₃
0.0 ppm

3d

ObsNuc
ObsFreq
Solvent
Reference

¹³C
150.9 MHz
CDCl₃
77.0 ppm

3d

ObsNuc
ObsFreq
Solvent
Reference

¹H
600.13 MHz
CDCl₃
0.0 ppm

3e

ObsNuc
ObsFreq
Solvent
Reference

¹³C
150.9 MHz
CDCl₃
77.0 ppm

3e

ObsNuc
ObsFreq
Solvent
Reference

¹H
600.13 MHz
CDCl₃
0.0 ppm

3f

ObsNuc
ObsFreq
Solvent
Reference

¹³C
150.9 MHz
CDCl₃
77.0 ppm

3f

ObsNuc
ObsFreq
Solvent
Reference

¹H
600.13 MHz
CDCl₃
0.0 ppm

3g

ObsNuc
ObsFreq
Solvent
Reference

¹³C
150.9 MHz
CDCl₃
77.0 ppm

3g

ObsNuc ¹H
ObsFreq 400.18 MHz
Frequency(Span) 8223.685 Hz
Scan 16
Temperature 22.85 °C
Solvent CDCl₃
Reference 0.0 ppm

2a

0.000

ObsNuc ^1H
ObsFreq 600.13 MHz
Frequency(Span) 12376.24 Hz
Scan 16
Temperature 24.85 °C
Solvent CDCl_3
Reference 0.0 ppm

2b

2b

ObsNuc ¹³C
ObsFreq 150.9 MHz
Frequency(Span) 35971.22 Hz
Scan 218
Temperature 24.85 °C
Solvent CDCl₃
Reference 77.0 ppm

ObsNuc
ObsFreq
Frequency(Span)
Scan
Temperature
Solvent
Reference

¹H
500.0 MHz
10000.0 Hz
8
22.7 °C
CDCl₃
0.0 ppm

2c

ObsNuc ¹³C
 ObsFreq 125.65 MHz
 Frequency(Span) 33898.3 Hz
 Scan 261
 Temperature 25.5 °C
 Solvent CDCl₃
 Reference 77.0 ppm

2c

ObsNuc	¹ H
ObsFreq	600.13 MHz
Frequency(Span)	8389.262 Hz
Scan	1024
Temperature	24.85 °C
Solvent	CDCl ₃
Reference	0.0 ppm

2d

2d

ObsNuc ¹³C
ObsFreq 150.9 MHz
Frequency(Span) 35971.22 Hz
Scan 10240
Temperature 25.85 °C
Solvent CDCl₃
Reference 77.0 ppm

ObsNuc
ObsFreq
Frequency(Span)
Scan
Temperature
Solvent
Reference

¹H
600.13 MHz
12376.24 Hz
16
24.85 °C
CDCl₃
0.0 ppm

2e

2e

ObsNuc ¹³C
ObsFreq 150.9 MHz
Frequency(Span) 35971.22 Hz
Scan 342
Temperature 24.85 °C
Solvent CDCl₃
Reference 77.0 ppm

ObsNuc ^1H
ObsFreq 270.05 MHz
Frequency(Span) 5405.4 Hz
Scan 8
Temperature 23.9 $^{\circ}\text{C}$
Solvent CDCl_3
Reference 0.0 ppm

2f

ObsNuc ¹³C
ObsFreq 67.8 MHz
Frequency(Span) 18348.6 Hz
Scan 256
Temperature 23.4 °C
Solvent CDCl₃
Reference 77.0 ppm

ObsNuc
ObsFreq
Frequency(Span)
Scan
Temperature
Solvent
Reference

¹H
600.13 MHz
12376.24 Hz
16
24.85 °C
CDCl₃
0.0 ppm

ObsNuc ¹³C
ObsFreq 150.9 MHz
Frequency(Span) 35971.22 Hz
Scan 203
Temperature 24.85 °C
Solvent CDCl₃
Reference 77.0 ppm

2g

ObsNuc
ObsFreq
Frequency(Span)
Scan
Temperature
Solvent
Reference

¹H
270.05 MHz
5405.4 Hz
8
23.2 °C
CDCl₃
0.0 ppm

2h

ObsNuc ¹³C
ObsFreq 150.9 MHz
Frequency(Span) 35971.22 Hz
Scan 118
Temperature 24.85 °C
Solvent ACETONE
Reference 206.0 ppm

2h

ObsNuc
ObsFreq
Frequency(Span)
Scan
Temperature
Solvent
Reference

¹H
270.05 MHz
5405.4 Hz
8
23.7 °C
CDCl₃
0.0 ppm

TMS

2i

ObsNuc 13C
ObsFreq 150.9 MHz
Frequency(Span) 35971.22 Hz
Scan 117
Temperature 24.85 °C
Solvent ACETONE
Reference 206.0 ppm

20.836
29.277
29.406
29.535
29.662
29.781
29.917
30.046
30.157

120.740
123.710
125.461
125.607
127.005
129.861
132.496
134.096
134.149
136.402

155.258
163.115
171.308
176.522

206.000

2i

ObsNuc ¹H
ObsFreq 270.05 MHz
Frequency(Span) 5405.4 Hz
Scan 8
Temperature 24.8 °C
Solvent CDCl₃
Reference 0.0 ppm

2j

2j

ObsNuc
ObsFreq
Frequency(Span)
Scan
Temperature
Solvent
Reference

¹³C
150.9 MHz
35971.22 Hz
170
24.85 °C
ACETONE
206.0 ppm

4b

ObsNuc ¹³C
ObsFreq 67.8 MHz
Freq (Span) 18348.6 Hz
Scan 256
Temperature 23.1 °C
Solvent CDCl₃
Reference 77.0 ppm

4c

ObsNuc ¹H
ObsFreq 270.05 MHz
Freq (Span) 5405.4 Hz
Scan 8
Temperature 19.7 °C
Solvent CDCl₃
Reference 0.0003 ppm

4c

ObsNuc
ObsFreq 67.8 MHz
Frequency(Span) 18348.6 Hz
Scan 128
Temperature 20.6 °C
Solvent CDCl₃
Reference 77.0 ppm

ObsNuc ¹H
ObsFreq 270.05 MHz
Freq (Span) 5405.4 Hz
Scan 8
Temperature 20.0 °C
Solvent CDCl₃
Reference 0.0 ppm

4d

ObsNuc ¹³C
ObsFreq 125.65 MHz
Frequency(Span) 33898.3 Hz
Scan 128
Temperature 23.6 °C
Solvent CDCl₃
Reference 77.0 ppm

4d

ObsNuc
ObsFreq
Frequency(Span)
Scan
Temperature
Solvent
Reference

¹H
270.05 MHz
5405.4 Hz
8
20.4 °C
CDCl₃
0.0 ppm

4e

ObsNuc
ObsFreq
Frequency(Span)
Scan
Temperature
Solvent
Reference

¹³C
125.65 MHz
33898.3 Hz
128
23.7 °C
CDCl₃
77.0 ppm

4e

ObsNuc ¹H
ObsFreq 600.13 MHz
Freq (Span) 12376.24 Hz
Scan 32
Temperature 24.85 °C
Solvent CDCl₃
Reference 0.0002 ppm

4f

4f

ObsNuc
ObsFreq 150.9 MHz
Frequency(Span) 35971.22 Hz
Scan 12038
Temperature 25.85 °C
Solvent CDCl₃
Reference 77.0 ppm

ObsNuc
ObsFreq 300.4 MHz
Frequency(Span) 6020.4 Hz
Scan 8
Temperature 21.0 °C
Solvent CDCl₃
Reference 0.0 ppm

4g

4g

ObsNuc ¹³C
ObsFreq 75.45 MHz
Frequency(Span) 20408.1 Hz
Scan 128
Temperature 20.5 °C
Solvent CDCl₃
Reference 77.0 ppm