**Efficient One-pot Synthesis of Highly Substituted Pyridin-2(1*H*)-ones
via the Vilsmeier-Haack Reaction of 1-Acetyl, 1-Carbamoyl
Cyclopropanes**

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I. General

All reagents were purchased from commercial sources and used without treatment, unless otherwise indicated. The products were purified by column chromatography over silica gel. ^1H NMR and ^{13}C NMR spectra were recorded at 25 °C on a Varian 500 MHz and 125 MHz, respectively, and TMS as internal standard. IR spectra (KBr) were recorded on a Magna-560 FTIR spectrophotometer in the range of 400~4000 cm^{-1} . Mass spectra were recorded on Agilent 1100 LCMsD mass spectrometer.

II. Synthesis and analytical data of 2a-g

General procedure for the preparation of **2** (with **2a** as an example): The Vilsmeier reagent was prepared by adding POCl_3 (5.0 mmol) dropwise to ice cold dry DMF (5 mL) under stirring. The mixture was then stirred for 10-15 min at 0 °C. To the above Vilsmeier reagent was added **1a** (1.0 mmol) as a solution in DMF (5.0 mL). Then the mixture was allowed to warm to 100 °C and was stirred for 1.0 h. After the starting material was consumed (monitored by TLC), the reaction mixture was poured into saturated sodium chloride aqueous (50 mL). The mixture was extracted with dichloromethane (3×20 mL), the combined organic phase was washed with water (3×20 mL), dried over MgSO_4 , filtered and concentrated in *vacuo*. The crude product was purified by flash chromatography (silica gel, petroleum ether: diethyl ether = 2: 1) to give **2a** (241 mg, 73%) as a white solid.

4-chloro-5-(2-chloroethyl)-1-(4-chlorophenyl)-6-oxo-1,6-dihydropyridine-3-carbaldehyde (2a): white solid; mp 154-156 °C ^1H NMR (CDCl_3 , 500 MHz) δ 3.30 (t, $J = 6.5$ Hz, 2H), 3.81 (t, $J = 7.0$ Hz, 2H), 7.32 (d, $J = 7.0$ Hz, 2H), 7.51 (d, $J = 7.0$ Hz, 2H), 8.11 (s, 1H), 10.16 (s, 1H). ^{13}C NMR (CDCl_3 , 125 MHz) δ 31.3, 41.2, 114.9, 127.4, 127.6 (2C), 129.8 (2C), 135.7, 137.5, 141.2, 145.0, 160.5, 186.4. IR (KBr, cm^{-1})

1645, 1549, 1540, 1440, 1392, 1082, 830. MS calcd m/z 330.5, found 369.1 $[M+39]^+$
 Anal. Calcd for $C_{14}H_{10}Cl_3NO_2$: C, 50.86; H, 3.05; N, 4.24. Found: C, 50.95; H, 3.11;
 N, 4.32.

4-chloro-5-(2-chloroethyl)-6-oxo-1-phenyl-1,6-dihydropyridine-3-carbaldehyde

(2b): white solid; mp 89-91 °C 1H NMR ($CDCl_3$, 500 MHz) δ 3.30 (t, $J = 7.5$ Hz, 2H), 3.80 (t, $J = 7.0$ Hz, 2H), 7.35 (d, $J = 7.5$ Hz, 2H), 7.49-7.53 (m, 3H), 8.15 (s, 1H), 10.14 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ 31.6, 41.5, 115.0, 126.5 (2C), 127.4, 129.8 (2C), 129.9, 139.4, 142.0, 145.1, 160.9, 186.8. IR (KBr, cm^{-1}) 1641, 1603, 1587, 1540, 1271. Anal. Calcd for $C_{14}H_{11}Cl_2NO_2$: C, 56.78; H, 3.74 ; N, 4.73. Found: C, 56.90; H, 3.82 ; N, 4.67.

4-chloro-5-(2-chloroethyl)-6-oxo-1-p-tolyl-1,6-dihydropyridine-3-carbaldehyde

(2c): white solid; mp 104-106 °C 1H NMR ($CDCl_3$, 500 MHz) δ 2.42 (s, 3H), 3.29 (t, $J = 7.0$ Hz, 2H), 3.80 (t, $J = 7.0$ Hz, 2H), 7.22 (d, $J = 6.5$ Hz, 2H), 7.31 (d, $J = 8.0$ Hz, 2H), 8.15 (s, 1H), 10.13 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ 21.2, 31.4, 41.2, 114.6, 125.8 (2C), 127.0, 130.2 (2C), 136.6, 139.8, 141.9, 144.7, 160.8, 186.5. IR (KBr, cm^{-1}) 1645, 1592, 1539, 1440, 1396, 1326, 1083, 821. Anal. Calcd for $C_{15}H_{13}Cl_2NO_2$: C, 58.08; H, 4.22; N, 4.52. Found: C, 50.79; H, 4.29; N, 4.46.

4-chloro-5-(2-chloroethyl)-1-(4-methoxyphenyl)-6-oxo-1,6-dihydropyridine-3-carbaldehyde (2d): white solid; mp 70-72 °C ^1H NMR (CDCl_3 , 500 MHz) δ 3.29 (t, $J = 7.5$ Hz, 2H), 3.79 (t, $J = 7.5$ Hz, 2H), 3.85 (s, 3H), 7.00 (d, $J = 9.0$ Hz, 2H), 7.26 (d, $J = 9.5$ Hz, 2H), 8.14 (s, 1H) 10.14 (s, 1H). ^{13}C NMR (CDCl_3 , 125 MHz) δ 30.1, 39.9, 54.4, 113.3, 113.4 (2C), 125.7, 126.0 (2C), 130.6, 140.7, 143.4, 158.9, 159.7, 185.3. IR (KBr, cm^{-1}) 1645, 1594, 1542, 1437, 1390, 1083, 834. Anal. Calcd for $\text{C}_{15}\text{H}_{13}\text{Cl}_2\text{NO}_3$: C, 55.23; H, 4.02; N, 4.29. Found: C, 55.18; H, 4.07; N, 4.38.

4-chloro-5-(2-chloroethyl)-6-oxo-1-o-tolyl-1,6-dihydropyridine-3-carbaldehyde (2e): white solid; mp 85-87 °C ^1H NMR (CDCl_3 , 500 MHz) δ 2.14 (s, 3H), 3.12 (t, $J = 7.0$ Hz, 2H), 3.83 (t, $J = 7.5$ Hz, 2H), 7.18 (d, $J = 8.0$ Hz, 1H), 7.34-7.38 (m, 2H), 7.41 (d, $J = 7.5$ Hz, 1H), 8.06 (s, 1H), 10.16 (s, 1H). ^{13}C NMR (CDCl_3 , 125 MHz) δ 17.8, 31.4, 41.5, 114.9, 126.9, 127.5, 127.7, 130.3, 131.7, 134.9, 138.7, 142.2, 145.1, 160.6, 186.8. IR (KBr, cm^{-1}) 1647, 1593, 1538, 1490, 1339, 1081, 747. Anal. Calcd for $\text{C}_{15}\text{H}_{13}\text{Cl}_2\text{NO}_2$: C, 58.08; H, 4.22; N, 4.52. Found: C, 58.16; H, 4.14; N, 4.46.

4-chloro-5-(2-chloroethyl)-1-(2-methoxyphenyl)-6-oxo-1,6-dihydropyridine-3-

carbaldehyde (2f): white solid; mp 126-128 °C ^1H NMR (CDCl_3 , 500 MHz) δ 3.31 (t, $J = 7.5$ Hz, 2H), 3.81 (t, $J = 7.5$ Hz, 2H), 3.82 (s, 3H), 7.07-7.11 (m, 2H), 7.25-7.28 (m, 1H), 7.48 (s, 1H), 8.05 (s, 1H), 10.14 (s, 1H). ^{13}C NMR (CDCl_3 , 125 MHz) δ 31.4, 41.2, 55.9, 112.3, 114.4, 121.0, 127.0, 127.7, 128.0, 131.3, 143.2, 144.6, 153.8, 160.6, 186.6. IR (KBr, cm^{-1}) 1643, 1589, 1539, 1439, 1387, 1085, 831. MS calcd m/z 326.0, found 326.0 $[\text{M}]^+$. Anal. Calcd for $\text{C}_{15}\text{H}_{13}\text{Cl}_2\text{NO}_3$: C, 55.23; H, 4.02; N, 4.29. Found: C, 55.29; H, 4.11; N, 4.38.

4-chloro-5-(2-chloroethyl)-1-(2,4-dimethylphenyl)-6-oxo-1,6-dihydropyridine-3-carbaldehyde (2g): white solid; mp 111-113 °C ^1H NMR (CDCl_3 , 500 MHz) δ 2.09 (s, 3H), 2.40 (s, 3H), 3.32 (t, $J = 7.0$ Hz, 2H), 3.82 (t, $J = 7.5$ Hz, 2H), 7.05 (d, $J = 8.0$ Hz, 1H), 7.15-7.18 (m, 2H), 8.05 (s, 1H), 10.15 (s, 1H). ^{13}C NMR (CDCl_3 , 125 MHz) δ 17.7, 21.4, 31.4, 41.5, 114.9, 126.7, 127.4, 128.2, 132.2, 134.4, 136.2, 140.4, 142.4, 145.0, 160.8, 186.7. IR (KBr, cm^{-1}) 1652, 1549, 1540, 1439, 1338, 1077, 787. Anal. Calcd for $\text{C}_{16}\text{H}_{15}\text{Cl}_2\text{NO}_2$: C, 59.28; H, 4.66; N, 4.32. Found: C, 59.37; H, 4.59; N, 4.38.

III. Synthesis and analytical data of 3a-g:

General procedure for the preparation of **3** (with **3a** as an example): The Vilsmeier reagent was prepared by adding POCl_3 (5.0 mmol) dropwise to ice cold dry DMF (5 mL) under stirring. The mixture was then stirred for 10-15 min at 0 °C. To the above Vilsmeier reagent was added **1a** (1.0 mmol) as a solution in DMF (5.0 mL). Then the mixture was allowed to warm to 120 °C and was stirred for 1.0 h. After the stirring material was consumed (monitored by TLC), the reaction mixture was poured into saturated sodium chloride aqueous (50 mL). The mixture was extracted with dichloromethane (3×20 mL), the combined organic phase was washed with water (3

× 20 mL), dried over MgSO₄, filtered and concentrated in *vacuo*. The crude product was purified by flash chromatography (silica gel, petroleum ether: diethyl ether = 2: 1) to give **3a** (235 mg, 78%) as a white solid.

4-chloro-3-(2-chloroethyl)-1-(4-chlorophenyl)pyridin-2(1H)-one (3a): white solid; mp 99-101 °C ¹H NMR (CDCl₃, 500 MHz) δ 3.24 (t, *J* = 7.0 Hz, 2H), 3.80 (t, *J* = 7.5 Hz, 2H), 6.36 (d, *J* = 7.5 Hz, 1H), 7.21 (d, *J* = 7.5 Hz, 1H), 7.32 (d, *J* = 9.0 Hz, 2H), 7.47 (d, *J* = 9.0 Hz, 2H). ¹³C NMR (CDCl₃, 125 MHz) δ 32.0, 41.7, 108.9, 127.8, 128.1 (2C), 129.8 (2C), 135.0, 135.4, 138.8, 146.0, 161.2. IR (KBr, cm⁻¹) 1641, 1604, 1589, 1461, 1407, 1326, 1095, 829. MS calcd *m/z* 302.0, found 302.0 [M]⁺. Anal. Calcd for C₁₃H₁₀Cl₃NO: C, 51.60; H, 3.33; N, 4.63. Found: C, 51.68; H, 3.25; N, 4.56.

Crystal data for **3a**: C₁₃H₁₀Cl₃NO, white crystal, $M = 302.57$, Monoclinic, P21/n, $a = 11.0773(4)$ Å, $b = 7.2082(3)$ Å, $c = 17.3552(6)$ Å, $\alpha = 90.00^\circ$, $\beta = 105.9840(10)^\circ$, $\gamma = 90.00^\circ$, $V = 1332.19(9)$ Å³, $Z = 4$, $T = 571(2)$, $F_{000} = 616$, $R_1 = 0.0418$, $wR_2 = 0.1076$. CCDC deposition number: 644180. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB21EZ, UK; fax: (+44)1223-336-033; or deposit@ccdc.cam.ac.uk).

4-chloro-3-(2-chloroethyl)-1-phenylpyridin-2(1H)-one (3b): white solid; mp 93-95 °C ¹H NMR (CDCl₃, 500 MHz) δ 3.24 (t, $J = 7.0$ Hz, 2H), 3.79 (t, $J = 7.0$ Hz, 2H), 6.33 (d, $J = 7.5$ Hz, 1H), 7.23-7.26 (m, 1H) 7.35 (d, $J = 7.5$ Hz, 2H), 7.42-7.45 (m, 1H), 7.48-7.51 (m, 2H). ¹³C NMR (CDCl₃, 125 MHz) δ 32.1, 41.8, 108.5, 126.7 (2C), 127.6, 129.0, 129.7 (2C), 135.9, 140.5, 145.7, 161.3. IR (KBr, cm⁻¹) 1643, 1585, 1540, 1433, 1325, 1078, 965. Anal. Calcd for C₁₃H₁₁Cl₂NO: C, 58.23; H, 4.13; N, 5.22. Found: C, 58.29; H, 4.22; N, 5.15.

4-chloro-3-(2-chloroethyl)-1-p-tolylpyridin-2(1H)-one (3c): white solid; mp 80-82 °C ¹H NMR (CDCl₃, 500 MHz) δ 2.41 (s, 3H), 3.23 (t, $J = 7.0$ Hz, 2H), 3.79 (d, $J = 7.0$ Hz, 2H), 6.31 (d, $J = 7.5$ Hz, 1H), 7.22-7.23 (m, 3H), 7.28-7.30 (m, 2H). ¹³C NMR (CDCl₃, 125 MHz) δ 21.4, 32.1, 41.8, 108.4, 126.4 (2C), 127.5, 130.2 (2C), 136.0, 138.0, 139.1, 145.6, 161.4. IR (KBr, cm⁻¹) 1647, 1587, 1541, 1439, 1389, 1081, 833. Anal. Calcd for C₁₄H₁₃Cl₂NO: C, 59.59; H, 4.64; N, 4.96. Found: C, 59.47; H, 4.59; N, 4.89.

4-chloro-3-(2-chloroethyl)-1-(4-methoxyphenyl)pyridin-2(1H)-one (3d): yellowish solid; mp 83-85 °C ^1H NMR (CDCl_3 , 500 MHz) δ 3.25 (t, $J = 7.0$ Hz, 2H), 3.71 (t, $J = 7.0$ Hz, 2H), 3.86 (s, 3H), 6.33 (d, $J = 7.5$ Hz, 1H), 7.03 (m, 2H), 7.24 (d, $J = 7.5$ Hz, 1H), 7.29 (d, $J = 8.5$ Hz, 2H). ^{13}C NMR (CDCl_3 , 125 MHz) δ 32.1, 41.8, 55.8, 108.4, 114.9, 122.2, 127.7, 129.2, 133.3, 136.2, 145.6, 159.8. IR (KBr, cm^{-1}) 1646, 1589, 1539, 1445, 1390, 1080, 833. Anal. Calcd for $\text{C}_{14}\text{H}_{13}\text{Cl}_2\text{NO}_2$: C, 56.39; H, 4.39; N, 4.70. Found: C, 56.33; H, 4.32; N, 4.63.

4-chloro-3-(2-chloroethyl)-1-o-tolylpyridin-2(1H)-one (3e): white solid; mp 76-78 °C ^1H NMR (CDCl_3 , 500 MHz) δ 2.13 (s, 3H), 3.24 (t, $J = 7.0$ Hz, 2H), 3.83 (t, $J = 7.5$ Hz, 2H), 6.33 (d, $J = 7.0$ Hz, 1H), 7.10 (d, $J = 7.0$ Hz, 1H), 7.16 (d, $J = 8.0$ Hz, 1H), 7.29-7.39 (m, 3H). ^{13}C NMR (CDCl_3 , 125 MHz) δ 17.8, 31.9, 41.8, 108.4, 122.6, 127.2, 127.4, 129.6, 131.4, 135.2, 135.9, 139.8, 145.8, 161.1. IR (KBr, cm^{-1}) 1648, 1590, 1542, 1441, 1388, 1081, 835. Anal. Calcd for $\text{C}_{14}\text{H}_{13}\text{Cl}_2\text{NO}$: C, 59.59; H, 4.64; N, 4.96. Found: C, 59.68; H, 4.57; N, 4.88.

4-chloro-3-(2-chloroethyl)-1-(2-methoxyphenyl)pyridin-2(1H)-one (3f): white

solid; mp 101-103 °C ^1H NMR (CDCl_3 , 500 MHz) δ 3.23 (t, $J = 7.0$ Hz, 2H), 3.78 (t, $J = 7.0$ Hz, 2H), 3.81 (s, 3H), 6.29 (d, $J = 7.5$ Hz, 1H), 7.03-7.07 (m, 2H), 7.10 (d, $J = 7.0$ Hz, 1H), 7.23 (d, $J = 8.0$ Hz, 1H), 7.40-7.43 (m, 1H). ^{13}C NMR (CDCl_3 , 125 MHz) δ 32.1, 41.8, 56.1, 108.0, 112.5, 121.2, 127.4, 128.6, 129.1, 130.8, 136.9, 145.7, 154.3, 161.3. IR (KBr, cm^{-1}) 1649, 1603, 1544, 1440, 1327, 1076, 740. MS calcd m/z 298.0, found 298.0 $[\text{M}]^+$. Anal. Calcd for $\text{C}_{14}\text{H}_{13}\text{Cl}_2\text{NO}_2$: C, 56.39; H, 4.39; N, 4.70. Found: C, 56.47; H, 4.27; N, 4.78.

4-chloro-3-(2-chloroethyl)-1-(2,4-dimethylphenyl)pyridin-2(1H)-one (3g): white solid; mp 90-92 °C ^1H NMR (CDCl_3 , 500 MHz) δ 2.07 (s, 3H), 2.36 (s, 3H), 3.23 (t, $J = 7.5$ Hz, 2H), 3.80 (t, $J = 7.5$ Hz, 2H), 6.30 (d, $J = 7.0$ Hz, 1H), 7.03 (d, $J = 8.0$ Hz, 1H), 7.08 (d, $J = 7.0$ Hz, 1H), 7.11-7.14 (m, 2H). ^{13}C NMR (CDCl_3 , 125 MHz) δ 17.7, 21.4, 31.9, 41.8, 108.3, 126.9, 127.6, 128.0, 132.1, 134.7, 136.1, 137.2, 139.5, 145.7, 161.2. IR (KBr, cm^{-1}) 1700, 1652, 1514, 1455, 671. Anal. Calcd for $\text{C}_{15}\text{H}_{15}\text{Cl}_2\text{NO}$: C, 60.83; H, 5.10; N, 4.73. Found: C, 60.75; H, 5.04; N, 4.66.

IV. Synthesis and analytical data of 4e

General procedure for the preparation of **4e**: The Vilsmeier reagent was prepared by adding POCl_3 (3.0 mmol) dropwise to ice cold dry DMF (5.0 mL) under stirring. The mixture was then stirred for 10-15 min at 0 °C. To the above Vilsmeier reagent was added **1e** (1.0 mmol) as a solution in DMF (5.0 mL). Then the mixture was allowed to warm to 80 °C and was stirred for 10 min. After the starting material was consumed (monitored by TLC), the reaction mixture was poured into saturated sodium chloride aqueous (50 mL). The mixture was extracted with dichloromethane (3×20 mL), the combined organic phase was washed with water (3×20 mL), dried over MgSO_4 , filtered and concentrated in *vacuo*. The crude product was purified by flash

chromatography (silica gel, petroleum ether: diethyl ether = 2: 1) to give **4e** (72 mg, 25%) as a white solid.

(Z)-3-chloro-2-(2-chloroethyl)-N-o-tolylbut-2-enamide (4e): white solid; mp 110-112 °C ^1H NMR (CDCl_3 , 500 MHz) δ 2.32 (s, 3H), 2.41 (s, 3H), 3.05 (t, $J = 6.5$ Hz, 2H), 3.80 (t, $J = 6.5$ Hz, 2H), 7.13-7.16 (m, 1H), 7.23-7.27 (m, 2H), 7.32 (brs, 1H), 7.88 (d, $J = 8.0$ Hz, 1H). ^{13}C NMR (CDCl_3 , 125 MHz) δ 17.9, 24.6, 34.7, 42.3, 122.8, 126.9, 129.0, 130.7, 131.6, 135.0, 136.5, 165.9. IR (KBr, cm^{-1}) 3029, 2339, 2170, 1628, 1533, 1486, 1384, 1321, 1112, 750. Anal. Calcd for $\text{C}_{13}\text{H}_{15}\text{Cl}_2\text{NO}$: C, 57.37; H, 5.56; N, 5.15. Found: C, 57.31; H, 5.49; N, 5.08.

V. Copies of NMR spectra for compounds 2a-g and 4e

new experiment
Archive directory: /export/home/pan/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: szpul
Solvent: CDCl3
Ambient temperature
INNOVA-500 "HREUS500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.999 sec
Width 18001.8 Hz
8 repetitions
OBSERVE H1, 499.8025909 MHz
DATA PROCESSING
F1 size 131072
Total time 0 min, 24 sec

STANDARD CARBON PARAMETERS
Archive directory: /export/home/fang/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: szpul
Solvent: CDCl3
Temp. 7.5 C / 280.6 K
User: 1-14-87
INNOVA-500 "HREUS500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.305 sec
Width 31421.6 Hz
120 repetitions
OBSERVE C13, 125.8754632 MHz
DECOUPLE H1, 499.8025909 MHz
Power 48 dB
on during acquisition
off during delay
WALTZ-16 modulated
Line broadening 1.0 Hz
F1 size 131072
Total time 3 hr, 12 min, 26 sec

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/lluy/vnmrsys/data
 Sample directory:
 File: PROTON
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 INOVA-500 "NENUS00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 10001.0 Hz
 8 repetitions
 OBSERVE H1: 499.0925000 MHz
 DATA PROCESSING
 FT size 85536
 Total time 0 min, 23 sec

2b

STANDARD CARBON PARAMETERS
 Archive directory: /export/home/lluy/vnmrsys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Temp: 25.0 C / 280.0 K
 User: 1-14-87
 INOVA-500 "NENUS00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.380 sec
 Width 31421.8 Hz
 64 repetitions
 OBSERVE C13: 125.6754316 MHz
 DECOUPLE H1: 499.0925000 MHz
 Power 40 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 3 hr, 17 min, 26 sec

2b

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/liuy/vnmrsys/data
 Sample directory:
 File: PROTON
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 INOVA-500 "MELNUS00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 10881.8 Hz
 8 repetitions
 OBSERVE F1: 499.0025309 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 23 sec

2c

STANDARD CARBON PARAMETERS
 Archive directory: /export/home/liuy/vnmrsys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Temp: 29.0 C / 293.1 K
 User: 1-14-07
 INOVA-500 "MELNUS00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.200 sec
 Width 31421.8 Hz
 2648 repetitions
 OBSERVE C13: 125.8754646 MHz
 DECOUPLE H1: 499.8058965 MHz
 Power 40 dB
 Continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 3 hr, 12 min, 26 sec

2c

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/lluy/vnmrsys/data
 Sample directory:
 File: PROTON
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 INOVA-500 "MENUM500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 10091.8 Hz
 OBSERVE H1, 499.002593 MHz
 8 repetitions
 DATA PROCESSING
 FT size 85536
 Total time 0 min, 23 sec

STANDARD CARBON PARAMETERS
 Archive directory: /export/home/lluy/vnmrsys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 User: 1-14-87
 INOVA-500 "MENUM500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.309 sec
 Width 31421.8 Hz
 OBSERVE C13, 125.0756252 MHz
 DECOUPLE H1, 499.002593 MHz
 Power 40 dB
 Continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 3 hr, 12 min, 26 sec

new experiment
 Archive directory: /export/home/fang/vnmr/sys/data
 Sample directory:
 File: PROTON
 Pulse Sequence: v2pul
 Solvent: CDCl3
 Ambient temperature
 INOVA-500 "NENUS00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 22002.2 Hz
 6 repetitions
 OBSERVE R1, 499.8025854 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 23 sec

STANDARD CARBON PARAMETERS
 Archive directory: /export/home/liuy/vnmr/sys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: v2pul
 Solvent: CDCl3
 Ambient temperature
 User: 1-14-07
 INOVA-500 "NENUS00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.308 sec
 Width 31421.8 Hz
 128 repetitions
 OBSERVE C13, 125.6754316 MHz
 DECOUPLE H1, 499.8050905 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 9 min, 51 sec

STANDARD PROTON PARAMETERS

Archive directory: /export/home/lluy/vmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENUS500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 18861.8 Hz
Observe F1: 499.8025029 MHz
8 repetitions
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec

2f

STANDARD CARBON PARAMETERS

Archive directory: /export/home/fang/vmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pu1
Solvent: CDCl3
Temp. 7.5 C / 280.6 K
Users: 1-14-05
INNOVA-500 "NENUS500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
Observe F1: 125.6754632 MHz
128 repetitions
DECOUPLE: C13, 125.6754632 MHz
Power 10 dB
on during acquisition
off during delay
WALTZ-16 modulated
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec

2f

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/liuy/vnmr/sys/data
 Sample directory:
 File: PROTON
 Pulse Sequence: szpu1
 Solvent: CDCl3
 Ambient temperature
 INOVA-500 "HREUS500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.852 sec
 Width 18001.8 Hz
 0 repetitions
 OBSERVE H1, 499.8025829 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 23 sec

STANDARD CARBON PARAMETERS
 Archive directory: /export/home/sunsg/vnmr/sys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: szpu1
 Solvent: CDCl3
 Ambient temperature
 User: 1-14-82
 INOVA-500 "HREUS500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 31421.0 Hz
 64 repetitions
 OBSERVE C13, 125.6754316 MHz
 DECOUPLE H1, 499.8059905 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 3 hr, 12 min, 26 sec

new experiment
 Archive directory: /export/home/fang/vnmrsys/data
 Sample directory:
 File: PROTON
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 INOVA-500 "HENU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.535 sec
 Width 22002.2 Hz
 6 repetitions
 OBSERVE H1, 499.8025854 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 23 sec

STANDARD CARBON PARAMETERS
 Archive directory: /export/home/fang/vnmrsys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 User: 1-14-87
 INOVA-500 "HENU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.309 sec
 Width 31421.8 Hz
 192 repetitions
 OBSERVE C13, 125.6754318 MHz
 DECOUPLE H1, 499.8050905 MHz
 Power 48.00
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 9 min, 51 sec

new experiment
 Archive directory: /export/home/pan/vnmrsys/data
 Sample directory:
 File: PROTON
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 INOVA-500 "MNU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.899 sec
 Width 18801.6 Hz
 8 repetitions
 OBSERVE H1, 499.9025909 MHz
 DATA PROCESSING
 FT size 131072
 Total time 0 min, 24 sec

STANDARD CARBON PARAMETERS
 Archive directory: /export/home/sunsg/vnmrsys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 User: 1-14-97
 INOVA-500 "MNU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.389 sec
 Width 31421.8 Hz
 64 repetitions
 OBSERVE C13, 125.6754316 MHz
 DECOUPLE H1, 499.8059395 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 3 hr, 12 min, 26 sec

new experiment
 Archive directory: /export/home/pan/vmrsys/data
 Sample directory:
 File: PROTON
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 INOVA-500 "NENUS00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.399 sec
 Width 18001.8 Hz
 8 repetitions
 OBSERVE H1 499.8025009 MHz
 DATA PROCESSING
 FT size 131072
 Total time 0 min, 24 sec

STANDARD CARBON PARAMETERS
 Archive directory: /export/home/sunsg/vmrsys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 User: l-14-87
 INOVA-500 "NENUS00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.399 sec
 Width 31421.8 Hz
 329 repetitions
 OBSERVE C13 125.0754316 MHz
 DECOUPLE H1 499.8050905 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 3 hr, 12 min, 26 sec

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/liuy/vnmrsys/data
 Sample directory:
 File: PROTON
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 INOVA-500 "MREUS00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 10001.0 Hz
 8 repetitions
 OBSERVE H1, 499.0025029 MHz
 DATA PROCESSING
 FT size 65536
 Total time 9 min, 23 sec

STANDARD CARBON PARAMETERS
 Archive directory: /export/home/liuy/vnmrsys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Temp. 7.5 C / 280.6 K
 User: 1-14-87
 INOVA-500 "MREUS00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.500 sec
 Width 31421.8 Hz
 128 repetitions
 OBSERVE C13, 125.6754316 MHz
 DECOUPLE H1, 499.0059305 MHz
 Power 40 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
 Line broadening 1.0 Hz
 FT size 131072
 Total time 3 hr, 12 min, 26 sec

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/liuy/vnmrsys/data
 Sample directory:
 File: PROTON
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 INOVA-500 "NENU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 18801.8 Hz
 8 repetitions
 OBSERVE H1, 499.8025929 MHz
 DATA PROCESSING
 FT size 45538
 Total time 0 min, 23 sec

STANDARD CARBON PARAMETERS
 Archive directory: /export/home/liuy/vnmrsys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Temp. 7.5 C / 280.6 K
 Users: 1-14-07
 INOVA-500 "NENU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.389 sec
 Width 31421.8 Hz
 64 repetitions
 OBSERVE C13, 125.6754316 MHz
 DECOUPLE H1, 499.8059905 MHz
 Power 40 dB
 on during acquisition
 off during delay
 WALTZ-16 modulated
 Line broadening 1.0 Hz
 DATA PROCESSING
 FT size 131972
 Total time 3 hr, 12 min, 26 sec

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/liuy/vnmrsys/data
 Sample directory:
 File: PROTON
 Pulse Sequence: szpul
 Solvent: CDCl3
 Ambient temperature
 INOVA-500 "MAGNUS"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 16891.0 Hz
 8 repetitions
 OBSERVE H1, 499.005500 MHz
 DATA PROCESSING
 FI size 8558
 Total time 8 min, 23 sec

STANDARD CARBON PARAMETERS
 Archive directory: /export/home/liuy/vnmrsys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: szpul
 Solvent: CDCl3
 Ambient temperature
 User: 1-14-8
 INOVA-500 "MAGNUS"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.309 sec
 Width 31431.0 Hz
 128 repetitions
 OBSERVE C13, 125.6754316 MHz
 DECOUPLE H1, 499.005000 MHz
 Power 48 dB
 Continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FI size 131072
 Total time 9 min, 51 sec

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 18001.8 Hz
8 repetitions
D5SRVF M1.488.8025938 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec

Solvent: CDCl₃
Temp. 7.5 C / 280.6 K
User: 1-14-87
INOVA-500 "NENU500"