Synthesis of multi ring-fused 2-pyridones via an acyl-ketene imine cyclocondensation

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Supporting Information

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Experimental section

General. All reactions were carried out under an inert atmosphere with dry solvents under anhydrous conditions, unless otherwise stated. 1,2-dichloroethane was distilled from calcium hydride and toluene distilled from sodium. TLC was performed on Silica Gel 60 F₂₅₄ (Merck) with detection by UV light and staining with a solution of Ce(SO₄)₂ 4 H₂O (2g), PMA (5g) and concentrated sulphuric acid (12 ml) in H₂O (188 ml). Flash column chromatography (eluents given in brackets) was performed on silica gel (Matrex, 60 Å, 35-70 µm, Grace Amicon). The ^1H and ^{13}C NMR spectra were recorded on a Bruker DRX-400 for solutions in CDCl₃ [residual CHCl₃ (δ_{H} 7.26 ppm) or CDCl₃ (δ_{C} 77.0 ppm) as internal standard] or (CD₃)₂SO [residual (CH₃)₂SO (δ_{H} 2.49 ppm) or (CD₃)₂SO (δ_{C} 40.0 ppm) as internal standard] at 298 K. Microwave reactions were performed using Smith Creator (Biotage, formerly Personal Chemistry) in a septa capped 2-5 mL SmithTM process vial with stirring.

Procedure for the preparation of 2-pyridones (**5a-d** and **9a-c**) using MWI:

Meldrum's acid derivative **1** (1.65 mmol) was added to a stirred solution of imine **4** (0.55 mmol) (or imine **8** for preparation of **9a-c**) solved in 1,2-dichloroethane (2.75 ml).

Trifluoroacetic acid (42 μ l, 0.55 mmol) was added and the mixture was irradiated at 160°C for 120s (fixed hold time, normal absorption level for compounds **5a-e** and high absorption level for compounds **9a-c**) and then cooled to room temperature. The resulting solution was diluted with CH_2Cl_2 and washed with sat $\text{NaHCO}_3(\text{aq})$, water and brine. The water phases were extracted with CH_2Cl_2 and the combined organic extracts were dried with Na_2SO_4 , filtered and concentrated. Purification by column chromatography gave **2-pyridones (5a-e) and (9a-c)**.

Procedure for the preparation of 2-pyridones using conventional heating: Meldrum's acid derivative **1** (1.5 mmol) was added to a stirred solution of imine **4** (0.5 mmol) solved in toluene (10 ml). Trifluoroacetic acid (0.5 mmol) was added and the solution was heated to reflux for 2h and then cooled to room temperature. The resulting solution was concentrated and then diluted with CH_2Cl_2 and washed with sat $\text{NaHCO}_3(\text{aq})$, water and brine. The water phases were extracted with CH_2Cl_2 and the combined organic extracts were dried with Na_2SO_4 , filtered and concentrated. Purification by column chromatography gave **2-pyridone (5c and 5d)**.

Procedure for the preparation of 2-pyridones (9d and e) using MWI:

Meldrum's acid derivative **7** (160 mg, 0.87 mmol) was added to a stirred solution of imine **8** (80mg, 0.44mmol) in 1,2-dichloroethane (2 ml). Trifluoroacetic acid (35 μ l) was added and the mixture was irradiated at 160°C for 12min (normal absorption level) and then cooled to 50°C and an additional portion of Meldrum's acid derivative **7** (80 mg, 0.44 mmol) was added. The mixture was then irradiated at 160°C for 11min (6min for compound **9d**) and then cooled room temperature. The resulting solution was diluted with CHCl_3 and washed with sat $\text{NaHCO}_3(\text{aq})$, and the water phase was extracted with CHCl_3 , the combined organic extracts were concentrated and purified by column chromatography [CHCl_3 :EtOAc] to give **2-pyridones (9 d and 9e)**.

1,3-oxazine-4-one (6).

Meldrum's acid derivative **1a** (1.8 mmol) was added to a stirred solution of imine **4a** (0.6 mmol) in toluene (12 ml). Triethylamine (1.2 mmol) was added and the solution was

heated to reflux. After 2h it was cooled to room temperature. and diluted with CH₂Cl₂ and washed with sat NaHCO₃(aq), water and brine. The water phases were extracted with CH₂Cl₂ and the combined organic extracts were dried with Na₂SO₄, filtered and concentrated. Purification by column chromatography [Heptane:EtOAc] gave **6** (177 mg, 86%) as a yellow solid.

¹H NMR (400 MHz, CDCl₃) δ 7.68–7.72 (m, 2H) 7.40-7.46 (m, 3H) 7.10 (s, 1H), 6.64 (s, 1H) 5.97 (s, 1H) 4.64-4.71 (m, 1H) 3.96 (s, 3H) 3.89 (s, 3H) 3.08-3.17 (m, 1H) 2.93-3.03 (m, 1H) 2.68-2.76 (m, 1H) 1.92 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 162.5, 160.5, 149.4, 148.1, 132.3, 131.0, 128.6, 128.6, 127.1, 125.9, 125.8, 110.8, 109.0, 96.3, 90.5, 56.3, 55.9, 36.2, 27.6, 23.1; HRMS (FAB+) Calcd. for [M+H]⁺ C₂₁H₂₂NO₄: 352.1549 Observed 352.1548

3,4-dihydroharmane (8)

POCl₃ (2ml) was added to a stirred solution of acetylated tryptamine (700 mg, 3.5 mmol) in MeCN (18 ml) this solution was then irradiated at 120°C for 300s (fixed hold time, normal absorption level) and then cooled to rt. Water (20 ml) was then added and the MeCN was removed under vacuum. The remaining water phase was made alkaline with a 10 % NaOH solution (pH ~11) and extracted with CH₂Cl₂ and the combined organic extracts were concentrated to yield **8** (610 mg, 96%) as a brown foam, which was used without further purification. Data in agreement with published data¹.

6,7-dihydro-9,10-dimethoxy-2-phenyl-benzo[a]quinolizine-4-one (5a); Data in agreement with published data².

6,7-dihydro-2-phenyl-benzo[a]quinolizine-4-one (5b). White solid.

¹H NMR (400 MHz, CDCl₃) δ 7.81 (dd, 1H, *J*₁ = 7.2 Hz, *J*₂ = 1.6 Hz), 7.63-7.67 (m, 2H), 7.34-7.51 (m, 5H) 7.30 (dd, 1H, *J*₁ = 7.3 Hz, *J*₂ = 1.4 Hz) 6.96 (d, 1H, *J* = 1.8 Hz) 6.83 (d, 1H, *J* = 1.8 Hz) 4.33 (t, 2H, *J* = 6.3 Hz), 3.03 (t, 2H, *J* = 6.3 Hz); ¹³C NMR (100 MHz, CDCl₃) δ 162.7, 151.1, 143.2, 138.1, 135.5, 130.2, 129.5, 129.4, 129.0, 128.0,

127.6, 126.8, 125.3, 115.3, 102.8, 39.2, 28.0; HRMS (FAB+) Calcd. for $[M+H]^+$ $C_{19}H_{16}NO$: 274.1232 Observed 274.1229.

6,7-dihydro-9,10-dimethoxy-2-methyl-benzo[a]quinolizine-4-one (5c). Data in agreement with published data²

6,7-dihydro-9,10-dimethoxy-2-cyclohexyl-benzo[a]quinolizine-4-one (5d). White solid; 1H NMR (400 MHz, $CDCl_3$) δ 7.15 (s, 1H), 6.72 (s, 1H), 6.39-6.42 (m, 1H), 6.34-6.37 (m, 1H), 4.22 (t, 2H, $J = 6.5$ Hz) 3.95 (s, 3H), 3.92 (s, 3H), 2.88 (t, 2H, $J = 6.5$ Hz), 2.28-2.43 (m, 1H), 1.67-1.96 (m, 5H), 1.17-1.47 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.0, 159.2, 150.8, 148.4, 142.4, 129.0, 121.8, 114.0, 110.4, 108.1, 102.6, 56.3, 56.0, 44.1, 39.0, 32.8, 27.6, 26.4, 25.9; HRMS (FAB+) Calcd. for $[M+H]^+$ $C_{21}H_{26}NO_3$: 340.1913 Observed 340.1920.

6,7-dihydro-9,10-dimethoxy-2-methyl-3-ethyl-benzo[a]quinolizine-4-one (5e). White solid; 1H NMR (400 MHz, $CDCl_3$) δ 7.13 (s, 1H), 6.71 (s, 1H), 6.38 (s, 1H), 4.26 (t, 2H, $J = 6.5$ Hz) 3.94 (s, 3H) 3.92 (s, 3H), 2.88 (t, 2H, $J = 6.5$ Hz) 2.65 (q, 2H, $J = 7.5$ Hz) 2.25 (s, 3H), 1.12 (t, 3H, $J = 7.5$ Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.0, 150.4, 148.3, 144.6, 139.1, 129.5, 128.6, 121.8, 110.4, 107.6, 105.0, 56.2, 56.0, 39.5, 27.8, 20.6, 19.3, 12.7; HRMS (FAB+) Calcd. for $[M+H]^+$ $C_{18}H_{22}NO_3$: 300.1600 Observed 300.1604.

6,7-dihydro-2-methyl-indolo[2,3-a]quinolizine-4-one (9a). Yellow solid; 1H NMR (400 MHz, $SO(CD_3)_2$) δ 11.61 (s, 1H), 7.57 (d, 1H, $J = 8.1$ Hz), 7.40 (d, 1H, $J = 8.2$ Hz), 7.18-7.23 (m, 1H), 7.03-7.08 (m, 1H), 6.54 (d, 1H, $J = 1.7$ Hz), 6.15-6.19 (m, 1H), 4.22 (t, 2H, $J = 6.9$ Hz), 3.02 (t, 2H, $J = 6.9$ Hz), 2.17 (s, 3H); ^{13}C NMR (100 MHz, $SO(CD_3)_2$) δ 161.9, 150.0, 138.6, 137.4, 128.1, 125.8, 124.3, 120.1, 119.8, 116.5, 113.6, 112.3, 102.4, 40.0, 21.3, 19.5; HRMS (FAB+) Calcd. for $[M+H]^+$ $C_{16}H_{15}N_2O$: 251.1184 Observed 251.1183

6,7-dihydro-2-cyclohexyl-indolo[2,3-*a*]quinolizine-4-one (9b). Yellow solid; ^1H NMR (400 MHz, $\text{SO}(\text{CD}_3)_2$) δ 11.60 (s, 1H), 7.57 (d, 1H, $J = 8.1$ Hz), 7.41 (d, 1H, $J = 8.2$ Hz), 7.18-7.24 (m, 1H), 7.03-7.08 (m, 1H), 6.68 (d, 1H, $J = 1.6$ Hz), 6.14 (d, 1H, $J = 1.6$ Hz), 4.21 (t, 2H, $J = 6.9$ Hz), 3.01 (t, 2H, $J = 6.9$ Hz), 2.30-2.40 (m, 1H) 1.68-1.85 (m, 5H) 1.15-1.46 (m, 5H); ^{13}C NMR (100 MHz, $\text{SO}(\text{CD}_3)_2$) δ 162.2, 158.8, 138.5, 137.6, 128.3, 125.8, 124.2, 120.0, 119.8, 114.2, 113.4, 112.2, 100.3, 43.7, 40.1, 32.6, 26.3, 26.0, 19.5; HRMS (FAB+) Calcd. for $[\text{M}+\text{H}]^+$ $\text{C}_{21}\text{H}_{23}\text{N}_2\text{O}$: 319.1810 Observed 319.1816.

6,7-dihydro-2-tetrahydrofuran-3-yl-indolo[2,3-*a*]quinolizine-4-one (9c). Yellow solid; ^1H NMR (400 MHz, CDCl_3) δ 9.61 (s, 1H), 7.58 (d, 1H, $J = 8.0$ Hz), 7.40 (d, 1H, $J = 8.2$ Hz) 7.27-7.30 (m, 1H), 7.13-7.19 (m, 1H), 6.50 (d, 1H, $J = 1.8$ Hz), 6.40-6.42 (m, 1H), 4.38-4.53 (m, 2H), 4.02-4.09 (m, 2H), 3.80-3.90 (m, 2H), 3.20-3.29 (quintet, 1H, $J = 7.1$ Hz), 3.11 (t, 2H, $J = 6.9$ Hz), 2.30-2.39 (m, 1H), 1.95-2.06 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 162.9, 155.1, 138.3, 138.0, 127.6, 125.8, 124.6, 120.5, 119.5, 115.5, 114.7, 111.7, 99.5, 73.2, 68.4, 44.3, 40.4, 33.3, 19.7 ; HRMS (FAB+) Calcd. for $[\text{M}+\text{H}]^+$ $\text{C}_{19}\text{H}_{19}\text{N}_2\text{O}_2$: 307.1447 Observed 307.1450.

6,7-dihydro-2-methyl-3-ethyl-indolo[2,3-*a*]quinolizine-4-one (9d). Yellow solid. ^1H NMR (400 MHz, $\text{SO}(\text{CD}_3)_2$) δ 11.56 (s, 1H), 7.55 (d, 1H, $J = 8.0$ Hz), 7.38 (d, 1H, $J = 8.0$ Hz), 7.18 (t, 1H, $J = 7.4$ Hz), 7.04 (t, 1H, $J = 7.4$ Hz) 6.53 (s, 1H) 4.25 (t, 2H, $J = 6.9$ Hz), 3.01 (t, 2H, $J = 6.9$ Hz), 2.51 (q, 2H, $J = 7.5$ Hz) 2.19 (s, 3H) 1.01 (t, 3H, $J = 7.5$ Hz); ^{13}C NMR (100 MHz, $\text{SO}(\text{CD}_3)_2$) δ 161.7, 144.5, 138.5, 134.4, 129.3, 128.3, 125.9, 124.0, 120.0, 119.6, 112.6, 112.2, 103.3, 40.7, 20.6, 19.6, 19.3, 13.3; HRMS (FAB+) Calcd. for $[\text{M}+\text{H}]^+$ $\text{C}_{18}\text{H}_{19}\text{N}_2\text{O}$: 279.1497 Observed 279.1496.

Dihydro-Sempervilam (9e)

^1H NMR (400 MHz, $\text{SO}(\text{CD}_3)_2$) δ 11.55 (s, 1H), 7.55 (d, 1H, $J = 7.9$ Hz), 7.38 (d, 1H, $J = 8.1$ Hz), 7.18 (t, 1H, $J = 7.4$ Hz), 7.04 (t, 1H, $J = 7.4$ Hz) 6.46 (s, 1H) 4.25 (t, 2H, $J = 6.9$ Hz), 3.00 (t, 2H, $J = 6.9$ Hz), 2.53-2.59 (m, 2H) 2.36-2.43 (m, 2H) 1.64-1.74 (m, 4H); ^{13}C NMR (100 MHz, $\text{SO}(\text{CD}_3)_2$) δ 161.8, 145.9, 138.4, 134.0, 128.4, 125.9, 124.7,

123.8, 119.9, 119.5 112.4, 112.1, 101.9, 40.4, 29.1, 24.3, 22.4, 22.1, 19.6; HRMS (FAB+) Calcd. for $[M+H]^+$ $C_{19}H_{19}N_2O$: 291.1497 Observed 291.1500.

References:

- (1) Hua, D. H.; Bharathi, S. N.; Panangadan, J. A. K.; Tsujimoto, A. *J. Org. Chem.* **1991**, 56, 6998-7007.
- (2) Roy, A.; Nandi, S.; Ila, H.; Junjappa, T. *Org. Lett.* **2001**, 3, 229-232.

S7

Current Data Parameters
NAME np121_2
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050803
Time 13.15
INSTRUM spect
PROBHD 5 mm TXO 13C/
PULPROG zgpg30
TD 44146
SOLVENT CDCl3
NS 429
DS 4
SWH 22075.055 Hz
FIDRES 0.500047 Hz
AQ 0.9999569 sec
RG 32768
DM 22.650 usec
DE 7.00 usec
TE 296.0 K
D1 0.00100000 sec
d11 0.03000000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 9.40 usec
PL1 -4.00 dB
SFO1 100.4724065 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 97.00 usec
PL2 29.00 dB
PL12 29.00 dB
SFO2 399.5319976 MHz

F2 - Processing parameters
SI 65536
SF 100.4619095 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 27.00 cm
F1P 214.522 ppm
F1 21551.29 Hz
F2P -5.214 ppm
F2 -523.77 Hz
PPMCH 8.13836 ppm/cm
HZCM 817.59473 Hz/cm

S8

Current Data Parameters
NAME PureLJ005a
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20040224
Time 3.01
INSTRUM spect
PROBHD 5 mm TXO 13C/
PULPROG zgpg30
TD 44146
SOLVENT CDCl3
NS 40000
DS 4
SWH 22075.055 Hz
FIDRES 0.500047 Hz
AQ 0.9399569 sec
RG 32768
DM 22.650 usec
DE 7.00 usec
TE 296.0 K
D1 0.00100000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.40 usec
PL1 -4.00 dB
SF01 100.4724065 MHz

===== CHANNEL f2 =====
CPOPRG2 waltz16
NUC2 1H
PCPD2 97.00 usec
PL2 20.00 dB
PL12 20.00 dB
SF02 399.5319976 MHz

F2 - Processing parameters
SI 65536
SF 100.4619038 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 27.00 cm
F1P 214.412 ppm
F1 21540.22 Hz
F2P -5.324 ppm
F2 -534.83 Hz
PPMCM 8.13836 ppm/cm
HZCM 817.59467 Hz/cm

S9

Current Data Parameters
 NAME np130h
 EXPNO 5
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050804
 Time 10.47
 INSTRUM spect
 PROBHD 5 mm TX0 13C/
 PULPROG zgdc30
 TD 44146
 SOLVENT CDC13
 NS 506
 DS 4
 SWH 22075.055 Hz
 FIDRES 0.500047 Hz
 AQ 0.999563 sec
 RG 32768
 DW 22.650 usec
 DE 7.00 usec
 TE 296.0 K
 D1 0.00100000 sec
 d11 0.03000000 sec

----- CHANNEL f1 -----
 NUC1 13C
 P1 9.40 usec
 PL1 -4.00 dB
 SF01 100.4724065 MHz

----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 PCPC2 57.00 usec
 PL2 20.00 dB
 PL12 20.00 dB
 SF02 399.5319975 MHz

F2 - Processing parameters
 SI 65536
 SF 100.4619051 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

10 NMR plot parameters
 CX 27.00 cm
 F1F 200.000 ppm
 F1 20092.38 Hz
 F2F -5.337 ppm
 F2 -536.18 Hz
 PPMCM 7.60508 ppm/cm
 HZCM 764.02059 Hz/cm

Current Data Parameters
 NAME np142
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050602
 Time 11.14
 INSTRUM spect
 PROBHD 5 mm TXO 13C/
 PULPROG zgpg30
 TO 44146
 SOLVENT CDCl3
 NS 5194
 DS 4
 SMH 22075.055 Hz
 FIDRES 0.500047 Hz
 AQ 0.999569 sec
 RG 32768
 ON 22.650 usec
 DE 7.00 usec
 TE 296.0 K
 D1 0.00100000 sec
 d11 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.40 usec
 PL1 -4.00 dB
 SFO1 100.4724065 MHz

===== CHANNEL f2 =====
 CPOPRG2 waltz16
 NUC2 1H
 PCPD2 97.00 usec
 PL2 20.00 dB
 PL12 20.00 dB
 SFO2 399.5319975 MHz

F2 - Processing parameters

SF 100.4619035 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

CX 27.00 cm
 F1P 200.000 ppm
 F1 20092.38 Hz
 F2P -5.320 ppm
 F2 -534.49 Hz
 PPMCM 7.60446 ppm/cm
 HZCM 763.95837 Hz/cm

S11

Current Data Parameters
NAME np133_2
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050922
Time 9.03
INSTRUM spect
PROBHD 5 mm TXO 13C/
PULPROG zgpg30
TD 44146
SOLVENT DMSO
NS 31202
DS 4
SWH 22075.055 Hz
FIDRES 0.500047 Hz
AQ 0.959569 sec
RG 32768
DW 22.650 usec
DE 7.00 usec
TE 295.0 K
D1 0.00100000 sec
d11 0.03000000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 9.40 usec
PL1 -4.00 dB
SFO1 100.4724055 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 97.00 usec
PL2 20.00 dB
PL12 20.00 dB
SFO2 399.5319976 MHz

F2 - Processing parameters
SI 65536
SF 100.4619005 MHz
WCM EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 27.00 cm
F1P 200.000 ppm
F1 20092.38 Hz
F2P -5.214 ppm
F2 -523.77 Hz
PPMCM 7.60050 ppm/cm
HZCM 763.56104 Hz/cm

S12

Current Data Parameters
NAME np135a
EXPNO 8
PROCNO 1

F2 - Acquisition Parameters

Date_ 20051003
Time 15.46
INSTRUM spect
PROBHD 5 mm TXO 13C/
PULPROG zgdc30
TD 44146
SOLVENT DMSO
NS 5206
DS 4
SWH 22075.055 Hz
FIDRES 0.500047 Hz
AQ 0.9999569 sec
RG 32768
DM 22.650 usec
DE 7.00 usec
TE 295.0 K
D1 0.00100000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 9.40 usec
PL1 -4.00 dB
SFO1 100.4724065 MHz

===== CHANNEL f2 =====

CPOPRG2 waltz16
NUC2 1H
PCPD2 97.00 usec
PL2 20.00 dB
PL12 20.00 dB
SFO2 399.5319976 MHz

F2 - Processing parameters

S1 65336
SF 100.4619002 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 27.00 cm
F1P 214.522 ppm
F1 21551.29 Hz
F2P -5.214 ppm
F2 -523.77 Hz
PPMCM 8.13936 ppm/cm
HZCM 817.59467 Hz/cm

ppm

162.23

158.84

138.49

137.59

128.30

125.79

124.21

120.04

119.78

114.19

113.36

112.19

100.30

43.67

40.63

40.42

40.21

40.00

39.79

39.58

39.37

32.60

26.34

26.03

19.49

9b

S13

Current Data Parameters
NAME np138a
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050805
Time 16.49
INSTRUM spect
PROBHD 5 mm TXO 13C/
PULPROG zgpg30
TD 44146
SOLVENT CDCl3
NS 1243
DS 4
SWH 22075.055 Hz
FIDRES 0.500047 Hz
AQ 0.9999569 sec
RG 32768
DW 22.650 usec
DE 7.00 usec
TE 296.0 K
D1 0.00100000 sec
d11 0.03000000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 9.40 usec
PL1 -4.00 dB
SFO1 100.4724065 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 97.00 usec
PL2 20.00 dB
PL12 20.00 dB
SFO2 399.5319976 MHz

F2 - Processing parameters
SI 65536
SF 100.4619041 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 27.00 cm
F1P 214.522 ppm
F1 21551.25 Hz
F2P -5.214 ppm
F2 -523.77 Hz
PPMCM 8.13836 ppm/cm
HZCM 817.59473 Hz/cm

S14

Current Data Parameters
NAME np167
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20051003
Time 7.56
INSTRUM spect
PROBHD 5 mm TXD 13C/
PULPROG zgpg30
TD 44146
SOLVENT DMSO
NS 19568
DS 4
SWH 22075.055 Hz
FIDRES 0.500047 Hz
AQ 0.9999569 sec
RG 32768
DN 22.650 usec
DE 7.00 usec
TE 296.0 K
D1 0.00100000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.40 usec
PL1 -4.00 dB
SFO1 100.4724065 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 97.00 usec
PL2 20.00 dB
PL12 20.00 dB
SFO2 399.5319976 MHz

F2 - Processing parameters
SI 65536
SF 100.4518965 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 27.00 cm
F1P 214.522 ppm
F1 21551.29 Hz
F2P -5.214 ppm
F2 -523.77 Hz
PPMCM 8.13836 ppm/cm
HZCM 817.59467 Hz/cm

S15

Current Data Parameters
NAME np174_2
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20051206
Time 8.29
INSTRUM spect
PROBHD 5 mm TXO 13C/
PULPROG zgpg30
TD 44146
SOLVENT DMSO
NS 36808
DS 4
SWH 22075.055 Hz
FIDRES 0.500047 Hz
AQ 0.9999569 sec
RG 32768
DM 22.650 usec
DE 7.00 usec
TE 296.0 K
D1 0.00100000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.40 usec
PL1 -4.00 dB
SF01 100.4724065 MHz

===== CHANNEL f2 =====
CPOPRG2 waltz16
NUC2 1H
PCPD2 97.00 usec
PL2 20.00 dB
PL12 20.00 dB
SF02 399.5319976 MHz

F2 - Processing parameters

SF 100.4619012 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 27.00 cm
F1P 214.522 ppm
F1 21551.29 Hz
F2P -5.214 ppm
F2 -523.77 Hz
PPMCM 8.13836 ppm/cm
HZCM 817.59467 Hz/cm