Efficient Method for the Synthesis of Polysubstituted Benzenes by One-Pot Tandem Reaction of Vinyl Malononitriles with Nitroolefins

Dong Xue^{*a}, Jie Li^a, Zun-Ting Zhang^a and Jin-Gen Deng^{*b}

^aKey Laboratory of Applied Surface and Colloid Chemistry (Shaanxi Normal University), Ministry of Education, School of Chemistry and Materials Science, Xi'an, 710062, People's Republic of China; ^bNational Engineering Research Center of Chiral Drugs and Key Laboratory of Asymmetric Synthesis and Chirotechnology of Sichuan Province, Chengdu Institute of Organic Chemistry, Chinese Academy of Sciences, Chengdu 610041, People's Republic of China

E-mail: xuedong_welcome@snnu.edu.cn, jgdeng@cioc.ac.cn

Supplementary Information

General experimental information.....	S2
One pot procedure for the synthesis of 4a-4m	S2
The copies of ¹ HNMR, ¹³ CNMR and HRMS spectra for compounds 3 , 4a-4m	S9

General experimental information

All starting materials were of the highest commercially available grade and used without further purification. All solvent were purchased from commercial sources and used without further purification. Reactions were monitored by thin layer chromatography using silica gel GF₂₅₄ plates. Column chromatography was performed on silica gel (300-400 mesh). NMR spectra were recorded on a 300 MHz instrument. ¹H NMR chemical shifts are reported in ppm (δ) relative to tetramethylsilane (TMS) with the solvent resonance employed as the internal standard (CDCl₃, δ 7.26 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet), coupling constants (Hz) and integration. ¹³C NMR chemical shifts are reported in ppm from tetramethylsilane (TMS) with the solvent resonance as the internal standard (CDCl₃, δ 77.0 ppm). IR data was obtained from F-T spectrometer and recorded as cm⁻¹. HRMS (ESI) performed on FT-ICR Mass Spectrum. M.P was determined in open capillaries and thermometer was not adjusted.

Experimental procedure

1. One pot procedure for the synthesis of 4a-4m:

A 5 mL of tube was charged with vinyl malononitriles (0.1 mmol), nitroolefins (0.1 mmol), 1 mL of CH₂Cl₂, and 1.4 μL of Et₃N (0.01 mmol). The solution was stirred at room temperature and monitored by TLC. After the reaction was completed, CH₂Cl₂ was removed under reduced pressure and 5 mL of CH₃CN and 6.8 mg of EtONa (0.1 mmol) was added. The mixture was refluxed and monitored by TLC. After the reaction was completed, the solvent was removed under reduced pressure. The product was purified by silica gel column chromatography (EtOAc/hexanes, 1/8) to yield the corresponding polysubstituted benzenes with the reaction time and yields showed in table 2.

Entry	Substrate	R ₃	Product	T (h) ^c (a ^d +b ^e)	Yield (%)
1	1a	Ph	4a	12 (10+2)	68
2	1b	Ph	4b	62 (60+2)	62
3	1c	Ph	4c	10 (8+2)	63
4	1a	CH ₃ CH ₂ CH ₂	4d	80 (77+3)	62
5	1a	<i>p</i> -MeO-Ph	4e	20 (18+2)	61
6	1d	Ph	4f	72 (70+2)	63
7	1e	Ph	4g	72 (70+2)	79
8	1a	Naphyl	4h	12 (10+2)	68
9	1f	Ph	4i	16 (14+2)	71
10	1g	Ph	4j	12 (10+2)	65
11	1h	Ph	4k	60 (57+3)	64
12	1i	Ph	4l	30 (28+2)	67
13	1j	Ph	4m	40 (37+3)	64

c: total reaction time for two steps; *d*: the reaction time of the first step; *e*: the reaction time of the second step.

4a: yellow solid; yield: 76%; mp: 204.2-205.8 °C; ¹HNMR (300Hz, CDCl₃) δ (ppm) 5.81 (s, 2H), 6.74 (s, 1H), 7.18-7.51 (m, 10H); ¹³CNMR (75Hz, CDCl₃) δ (ppm) 149.5, 144.8, 142.9, 137.3, 137.0,

129.8, 128.9, 128.5, 127.2, 121.2, 115.8, 97.8; IR (KBr): 3469, 3363, 2923, 2214, 1619, 1559, 1497, 1445, 1328, 1277, 1076, 865, 821, 770, 698, 636 cm^{-1} ; HRMS (ESI) ($\text{M}+\text{Na}$)⁺: $\text{C}_{19}\text{H}_{13}\text{N}_3\text{O}_2+\text{Na}$, calculated: 338.0900, found: 338.0898.

4b: 3-amino-5-methyl-2-nitrobiphenyl-4-carbonitrile: Yellow solid;

yield: 62%; mp: 162.5-164.5 °C; ¹HNMR (300Hz, CDCl_3) δ (ppm) 2.53 (s, 3H), 5.77 (s, 2H), 6.61 (s, 1H), 7.28-7.41 (m, 5H); ¹³CNMR (75Hz, CDCl_3) δ (ppm) 147.2, 144.3, 142.9, 137.5, 128.9, 128.7, 127.1, 121.6, 115.2, 21.2; IR

(KBr): 3439, 3367, 3261, 2358, 2213, 1652, 1597, 1557, 1519, 1365, 1281, 842, 755, 701 cm^{-1} ; HRMS (ESI) ($\text{M}+\text{NH}_4$)⁺: calculated: 271.1190, found: 271.1191.

4c: 2-amino-3-nitro-4-phenyl-5,6,7,8-tetrahydronaphthalene-1-carbonitrile: Yellow

solid; yield: 62%; mp: 211.2-214.3 °C; ¹HNMR (300Hz, CDCl_3) δ (ppm) 1.57 (t, $J = 5\text{Hz}$, 2H), 1.73 (t, $J = 5.16\text{Hz}$, 2H), 2.16 (t, $J = 6\text{Hz}$,

2H), 2.89 (t, $J = 6\text{Hz}$, 2H), 5.26 (s, 2H), 7.06-7.35 (m, 5H); ¹³CNMR (75Hz, CDCl_3) δ (ppm) 146.0, 141.5, 141.3, 135.6, 128.6, 128.3, 127.9, 126.8, 115.2, 99.2, 29.5, 27.5, 22.5, 21.7; IR (KBr): 3464, 3357, 2940, 2221, 1641, 1589, 1546, 1516, 1443, 1363, 1335, 1264, 1169, 818, 773, 701 cm^{-1} ; HRMS (ESI) ($\text{M}+\text{Na}$): $\text{C}_{17}\text{H}_{15}\text{N}_3\text{O}_2+\text{Na}$, calculated: 316.1056, found: 316.1054.

4d: 3-amino-4-nitro-5-propylbiphenyl-2-carbonitrile: Yellow

solid; yield: 63%; mp: 113.7-115.2 °C; ¹HNMR (300Hz, CDCl_3) δ (ppm) 1.00 (t, $J = 7.2\text{Hz}$, 3H), 1.68 (m, 2H), 2.78 (t, $J = 7\text{Hz}$, 2H),

5.83 (s, 2H), 6.71 (s, 1H), 7.49-7.53 (m, 5H); ¹³C NMR (75Hz, CDCl_3) δ (ppm) 145.2, 144.3, 129.7, 128.9, 128.4, 120.8, 116.0, 35.9, 23.7, 14.1; IR (KBr): 3479, 3385, 2965,

2212, 1625, 1591, 1564, 1514, 1448, 1339, 1283, 838, 771 cm^{-1} ; HRMS (ESI) $(\text{M}+\text{Na})^+$:

$\text{C}_{16}\text{H}_{15}\text{N}_3\text{O}_2+\text{Na}$, calculated: 304.1062, found: 304.1103.

4e: Yellow solid; yield: 61%; mp: 167.2-169.3 $^{\circ}\text{C}$; ^1H NMR

(300Hz, CDCl_3) δ (ppm) 3.84 (s, 3H), 5.77 (s, 2H), 6.79 (s, 1H),

6.95 (d, $J = 8.4\text{Hz}$, 2H), 7.27 (d, $J = 8.4\text{Hz}$, 2H), 7.50-7.56 (m,

5H); ^{13}C NMR (75Hz, CDCl_3) δ (ppm) 160.4, 149.2, 144.7, 142.4, 137.2, 132.0, 129.3,

128.9, 128.6, 123.0, 122.3, 114.7, 97.2, 55.4; IR (KBr): 3469, 3356, 2928, 2837, 2215,

1617, 1558, 1506, 1454, 1253, 1174, 1028, 830, 775, 700 cm^{-1} ; HRMS (ESI) $(\text{M}+\text{Na})^+$:

$\text{C}_{20}\text{H}_{15}\text{N}_3\text{O}_3+\text{Na}$, calculated: 368.1006, found: 368.1004.

4f: 3-amino-5-(naphthalen-1-yl)-2-nitro-1-phenyl-4-cyanobenzene:

Yellow solid; Yield: 63%; mp: 185.2-185.4 $^{\circ}\text{C}$; ^1H NMR (300Hz,

CDCl_3) δ (ppm) 5.84 (s, 2H), 6.86 (s, 1H), 7.37-8.06 (m, 12H);

^{13}C NMR (75Hz, CDCl_3) δ (ppm) 129.0, 128.7, 127.3, 127.1, 127.1,

126.5, 125.2, 124.9, 122.7; IR (KBr): 3446, 3342, 3236, 3055, 2360, 2222, 1634, 1586,

1557, 1509, 1418, 1280, 841, 802, 778, 701 cm^{-1} ; HRMS (ESI) $(\text{M}+\text{Na})^+$: $\text{C}_{23}\text{H}_{15}\text{N}_3\text{O}_2+\text{Na}$,

calculated: 388.1056, found: 388.1051.

4g: 3-amino-5-(naphthalen-2-yl)-2-nitro-1-phenyl-4-cyanobenzene:

Yellow solid; Yield: 79%; mp: 178.5-181.5 $^{\circ}\text{C}$; ^1H NMR (300Hz,

CDCl_3) δ (ppm) 5.91 (s, 2H), 6.92 (s, 1H), 7.35-8.07 (m, 12H);

^{13}C NMR (75Hz, CDCl_3) δ (ppm) 144.9, 134.3, 129.0, 128.8, 128.6, 128.4, 127.8, 127.4,

127.3, 127.0, 125.5, 121.5; IR (KBr): 3465, 3347, 3028, 2362, 2219, 1621, 1584, 1556,

1495, 1442, 1327, 1272, 850, 818, 752, 697 cm^{-1} ; HRMS (ESI) $(\text{M}+\text{Na})^+$: $\text{C}_{23}\text{H}_{15}\text{N}_3\text{O}_2+\text{Na}$, calculated: 388.1056, found: 388.1059.

4h:3-amino-5-(naphthalen-2-yl)-4-nitrobiphenyl-2-carbonitri

le: Yellow solid; Yield: 68%; mp: 183.2-185.3 $^{\circ}\text{C}$; ^1H NMR (300Hz, CDCl_3) δ (ppm) 6.05 (s, 2H), 6.79 (s, 1H), 7.28-7.84 (m,

12H); ^{13}C NMR (75Hz, CDCl_3) δ (ppm) 149.6, 145.2, 142.2, 136.9, 135.3, 135.3, 133.5, 130.8, 129.9, 129.0, 128.9, 128.6, 128.5, 126.9, 126.3, 125.3, 125.2, 124.5, 122.2, 115.8; IR (KBr): 3457, 3348, 3235, 2218, 1634, 1594, 1558, 1513, 1281, 862, 803, 776, 698 cm^{-1} ; HRMS (ESI) $(\text{M}+\text{Na})^+$: $\text{C}_{23}\text{H}_{15}\text{N}_3\text{O}_2+\text{Na}$, calculated: 388.1056, found: 388.1055.

4i:3-amino-2-nitro-1-phenyl-9,10-dihydrophenanthrene-4-carbo

nitrile: Yellow solid; Yield: 71%; mp: 201.2-203.4 $^{\circ}\text{C}$; ^1H NMR (300Hz, CDCl_3) δ (ppm) 2.39 (t, $J = 6\text{Hz}$, 2H), 2.64 (t, $J = 6\text{Hz}$, 2H),

5.66 (s, 2H), 7.19-7.45 (m, 8H), 8.24 (d, $J = 7.3\text{Hz}$, 1H); ^{13}C NMR (75Hz, CDCl_3) δ (ppm) 143.3, 140.3, 139.8, 135.7, 131.1, 130.5, 128.8, 128.6, 128.2, 128.0, 127.9, 127.4, 127.1, 117.2, 29.1, 25.9; IR (KBr): 3439, 3367, 3261, 2213, 1652, 1597, 1557, 1519, 1430, 1365, 1281, 842, 755, 701 cm^{-1} ; HRMS (ESI) $(\text{M}+\text{Na})^+$: $\text{C}_{21}\text{H}_{15}\text{N}_3\text{O}_2+\text{Na}$, calculated: 364.1056, found: 364.1053.

4j:3-amino-7-methoxy-2-nitro-1-phenyl-9,10-dihydrophenant

hrene-4-carbonitrile: Yellow solid; Yield: 65%; mp: 185.6-187.8 $^{\circ}\text{C}$; ^1H NMR (300Hz, CDCl_3) δ (ppm) 2.37 (t, $J =$

6Hz, 2H), 2.61 (t, $J = 6\text{Hz}$, 2H), 3.87 (s, 3H), 5.71 (s, 2H), 6.79 (s, 1H), 6.93 (dd, $J =$ 8.4Hz, 2.1Hz, 1H), 7.19-7.44 (m, 5H), 8.20 (d, $J = 8.4\text{Hz}$, 1H); ^{13}C NMR (75Hz, CDCl_3) δ

(ppm) 161.4, 143.5, 142.0, 129.2, 128.7, 128.4, 128.0, 127.4, 117.4, 113.7, 112.1, 55.4, 29.5, 25.9; IR (KBr): 3470, 3356, 2933, 2833, 2360, 2207, 1615, 1586, 1541, 1495, 1415, 1333, 1266, 1213, 1034, 722, 702 cm^{-1} ; HRMS (ESI) $(\text{M}+\text{Na})^+$: $\text{C}_{22}\text{H}_{17}\text{N}_3\text{O}_3+\text{Na}$, calculated: 394.1162, found: 394.1167.

4k: 9-amino-2,3-dimethyl-8-nitro-7-phenyl-6H-benzo[c]chrome

ne-10-carbonitrile: Yellow solid; Yield: 64%; mp: 207.5-209.5

$^{\circ}\text{C}$; ^1H NMR (300Hz, CDCl_3) δ (ppm) 2.28 (s, 3H), 2.32 (s, 3H), 4.56 (s, 2H), 5.87 (s, 2H), 6.81 (s, 1H), 7.16-7.45 (m, 5H), 8.15 (s, 1H); ^{13}C NMR (75Hz, CDCl_3) δ (ppm) 154.9, 144.7, 142.8, 139.0, 137.8, 134.3, 131.0, 128.9, 127.7, 127.0, 122.3, 118.5, 117.7, 116.8, 65.6, 20.1, 19.3; IR (KBr): 3476, 3362, 2921, 2853, 2361, 2215, 1620, 1592, 1547, 1495, 1435, 1343, 1257, 1101, 1026, 926, 881, 781, 755, 702 cm^{-1} ; HRMS (ESI) $(\text{M}+\text{Na})^+$: $\text{C}_{22}\text{H}_{17}\text{N}_3\text{O}_3+\text{Na}$, calculated: 394.1168, found: 394.1167.

4l: 3-amino-5-(furan-2-yl)-2-nitrobiphenyl-4-carbonitrile:

Yellow solid; yield: 67%; mp: 188.4-191.2 $^{\circ}\text{C}$; ^1H NMR (300Hz, CDCl_3) δ (ppm) 5.96 (s, 2H), 6.61 (s, 1H), 7.19-7.71 (m, 8H); ^{13}C

NMR (75Hz, CDCl_3) δ (ppm) 148.7, 145.4, 144.8, 143.5, 137.6, 136.2, 128.9, 127.2, 117.1, 116.2, 113.3, 112.8; IR (KBr): 3468, 3364, 3149, 2361, 2212, 1617, 1553, 1497, 1438, 1275, 1248, 1029, 757, 698 cm^{-1} ; HRMS (ESI) $(\text{M}+\text{Na})^+$: $\text{C}_{17}\text{H}_{11}\text{N}_3\text{O}_3+\text{Na}$, calculated: 328.0693, found: 328.0695.

4m: 3-amino-2-nitro-5-(thiophen-2-yl)biphenyl-4-carbonitrile:

Yellow solid; Yield: 64%; mp: 200.4-202.2 $^{\circ}\text{C}$; ^1H NMR (300Hz, CDCl_3) δ (ppm): 5.94 (s, 2H), 6.91 (s, 1H), 7.17-7.76 (m, 8H); ^{13}C

NMR (75Hz, CDCl₃) δ (ppm): 145.4, 143.3, 141.3, 137.3, 139.0, 128.6, 127.2, 120.6, 116.2; IR (KBr): 3465, 3360, 3102, 2922, 2212, 1617, 1533, 1492, 1421, 1328, 1275, 1244, 850, 762, 704 cm⁻¹; HRMS (ESI) (M+Na)⁺: C₁₇H₁₁N₃O₂S+Na, calculated: 344.0464, found: 344.0463.

2. The copies of ^1H NMR, ^{13}C NMR and HRMS spectras of compounds 3, 4a-4m:

¹HNMR, in CDCl₃
Table 1 entry 17

Current Data Parameters
NAME xuedong2006snh
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060313
Time 17.24
INSTRUM av300
PROBHD 5 mm BBI 1H-
PULPROG zg
TD 32768
SOLVENT CDCl3
VS 27
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 181
DM 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 7.70 usec
PL1 -2.00 dB
SF01 300.1321009 MHz

F2 - Processing parameters
SI 16384
SF 300.1300291 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D F1F2 plot parameters
CX 20.00 cm
CY 10.00 cm
P1P 9.000 ppm
F1 2701.17 Hz
F2P -1.000 ppm
F2 -300.13 Hz
PPHMM 0.50000 ppm/cm
GZCM 150.06502 Hz/cm

Current Data Parameters
NAME xadong2006s-h
EXPNO 19
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060511
Time 17.23
INSTRUM av300
PROBHD 5 mm BBI 1H-3B
PULPROG zg
TD 32756
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.162956 Hz
AQ 2.7329011 sec
RG 256
CH 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.30000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -2.00 dB
SF01 300.1321009 MHz

F2 - Processing parameters
SI 16384
SF 300.1300078 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

JD NMR plot parameters
CX 20.00 cm
CY 7.00 cm
FIP 12.000 ppm
FI 3501.96 Hz
FQP -1.000 ppm
F2 -300.13 Hz
PPHMC 0.65000 ppm/cm
HZCM 195.08450 Hz/cm

Current Data Parameters
NAME xadong2006s-h
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060519
Time 20.27
INSTRUM av300
PROBHD 5 mm BBI 1H-13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 0
SWH 15115.541 Hz
FIDRES 0.275427 Hz
AQ 1.0060436 sec
RG 203.2
CH 27.600 usec
DE 5.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.02000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 20.50 usec
PL1 0.00 dB
SF01 75.4752558 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 20.00 dB
PL12 17.50 dB
SF02 300.1300004 MHz

F2 - Processing parameters
SI 131072
SF 75.4677460 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

JD NMR plot parameters
CX 20.00 cm
CY 14.00 cm
FIP 220.000 ppm
FI 15502.90 Hz
FQP 0.200 ppm
F2 0.00 Hz
PPHMC 11.00000 ppm/cm
HZCM 830.14520 Hz/cm

Current Data Parameters
NAME: kuedongsnc
EXPNO: 2
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20060515
Time: 15.19
INSTRUM: av300
PROBHD: 5 mm BBI 1H-8B
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 2048
DS: 0
SWH: 18115.941 Hz
FIDRES: 0.276427 Hz
AQ: 1.8089436 sec
RG: 71.8
DM: 27.600 usec
DE: 6.00 usec
TE: 300.0 K
D1: 2.00000000 sec
d11: 0.03000000 sec

----- CHANNEL f1 -----
NUC1: 13C
P1: 10.50 usec
PL1: -6.00 dB
SFO1: 75.4752958 MHz

----- CHANNEL f2 -----
CPOPRG2: waltz16
NUC2: 1H
PCPD2: 80.00 usec
PL2: 20.50 dB
PL12: 17.50 dB
SFO2: 300.1305004 MHz

F2 - Processing parameters
SI: 131072
SF: 75.4677490 MHz
RGW: 64
SSB: 0
LB: 3.00 Hz
GB: 0
PC: 1.40

ID NMR plot parameters
CX: 20.00 cm
CY: 14.00 cm
FIP: 200.000 ppm
F1: 16602.90 Hz
F2P: 0.000 ppm
F2: 0.00 Hz
PPHMC: 11.00000 ppm/cm
HZCM: 830.14520 Hz/cm

Current Data Parameters
NAME: xuecong2006snh
EXPNO: 36
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20061029
Time: 14.23
INSTRUM: av300
PROBHD: 5 mm BBI 1H-BB
PULPROG: zgpg30
TD: 32768
SOLVENT: CDCl3
NS: 4
DS: 4
SWH: 5995.204 Hz
FIDRES: 0.162959 Hz
AQ: 2.7329011 sec
RG: 574.7
CW: 83.400 usec
DE: 5.00 usec
TE: 300.2 K
D1: 2.0000000 sec

===== CHANNEL f1 =====
NUC1: 1H
P1: 7.70 usec
PL1: -2.00 dB
SFO1: 300.1321009 MHz

F2 - Processing parameters
SI: 16384
SF: 300.1300088 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.00

1D NMR plot parameters
CX: 20.00 cm
CY: 10.00 cm
F1P: 10.000 ppm
F1: 3001.30 Hz
F2P: -1.000 ppm
F2: -300.13 Hz
PCHW: 0.85000 ppm/cm
HZCM: 165.67150 Hz/cm

Current Data Parameters
NAME: xuecong2006snh
EXPNO: 8
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20061031
Time: 15.50
INSTRUM: av300
PROBHD: 5 mm BBI 1H-BB
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 714
DS: 0
SWH: 18115.341 Hz
FIDRES: 0.276527 Hz
AQ: 1.8005436 sec
RG: 35.9
CW: 27.500 usec
DE: 6.00 usec
TE: 302.0 K
D1: 2.0000000 sec
D11: 0.0300000 sec

===== CHANNEL f1 =====
NUC1: 13C
P1: 10.50 usec
PL1: -6.00 dB
SFO1: 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2: waltz15
NUC2: 1H
PCPD2: 20.00 usec
PL2: 20.60 dB
PL12: 17.50 dB
SFO2: 300.1359004 MHz

F2 - Processing parameters
SI: 131072
SF: 75.4677490 MHz
WDW: EM
SSB: 0
LB: 2.00 Hz
GB: 0
PC: 1.40

1D NMR plot parameters
CX: 20.00 cm
CY: 7.00 cm
F1P: 220.300 ppm
F1: 15692.90 Hz
F2P: 0.000 ppm
F2: 0.00 Hz
PCHW: 11.00000 ppm/cm
HZCM: 830.14520 Hz/cm

Current Data Parameters
NAME xuedong2005enh
EXPNO 30
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061016
Time 15:04
INSTRUM av300
PROBHD 5 mm BBI 1H-6B
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 574.7
OW B3 400 usec
DE 8.00 usec
TE 300.0 K
D1 2.30000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -2.00 dB
SFO1 300.1321009 MHz

F2 - Processing parameters
SI 15384
SF 300.1302320 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -1.000 ppm
F2 -300.13 Hz
PRCM 0.55300 ppm/cm
HZCM 155.07152 Hz/cm

Current Data Parameters
NAME xuedong2005enh
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061018
Time 17:52
INSTRUM av300
PROBHD 5 mm BBI 1H-6B
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 0
SWH 19115.941 Hz
FIDRES 0.276427 Hz
AQ 1.8068436 sec
RG 32
OW 27.500 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PL1 -6.00 dB
SFO1 75.4752958 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 20.50 dB
PL12 17.50 dB
SFO2 300.1302320 MHz

F2 - Processing parameters
SI 131072
SF 75.4677430 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 5.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -20.000 ppm
F2 -1509.36 Hz
PRCM 11.00000 ppm/cm
HZCM 170.14926 Hz/cm

Current Data Parameters
NAME xuedong2006snh
EXPNO 9
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060327
Time 17.53
INSTRUM av300
PROBHD 5 mm BBI 1H-BB
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 512
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
C1 2.0000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -2.00 dB
SFO1 300.1321009 MHz

F2 - Processing parameters
SI 16384
SF 300.1300073 MHz
WDW EM
SSB 0
LB 1.00 Hz
RB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 9.002 ppe
F1 2701.17 Hz
F2P -1.000 ppm
F2 -300.13 Hz
PPHCH 0.50000 ppm/cm
HZCM 150.06500 Hz/cm

Current Data Parameters
NAME xuedong2006snh
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060331
Time 20.29
INSTRUM av300
PROBHD 5 mm BBI 1H-BB
PULPROG zgpgc
TD 65536
SOLVENT CDCl3
NS 2048
DS 0
SWH 16115.941 Hz
FIDRES 0.276427 Hz
AQ 1.8028436 sec
RG 456.1
DW 27.600 usec
DE 6.00 usec
TE 300.0 K
C1 2.0000000 sec
d11 0.0300000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PL1 -6.00 dB
SFO1 75.4750505 MHz

===== CHANNEL f2 =====
CPDPRG2 gprg
NUC2 1H
PCPD2 80.00 usec
PL2 20.60 dB
PL12 17.50 dB
SFO2 300.1326622 MHz

F2 - Processing parameters
SI 131072
SF 75.4677490 MHz
WDW EM
SSB 0
LB 3.00 Hz
SB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 220.000 ppe
F1 15602.90 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPHCH 11.90000 ppm/cm
HZCM 830.14520 Hz/cm

Current Data Parameters
NAME xuedong2006sh
EXPNO 16
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060419
Time 21.31
INSTRUM av300
PROBHD 5 mm BBI 1H-8B
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 2048
DS 0
SWH 18115.941 Hz
FIDRES 0.276427 Hz
AQ 1.8088435 sec
RG 90.5
OW 27.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec
d11 0.0300000 sec

===== CHANNEL f1 =====
NUC1 ^{13}C
P1 10.50 usec
PL1 -6.00 dB
SFO1 75.4752958 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 80.00 usec
PL2 20.50 dB
PL12 17.50 dB
SFO2 300.1309004 MHz

F2 - Processing parameters
SI 131072
SF 75.4677450 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PHCM 19.30000 ppm/cm
HZCM 754.67755 Hz/cm

Current Data Parameters
NAME xuedong2006snh
EXPNO 42
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061102
Time 10.49
INSTRUM av300
PROBHD 5 mm BBI 1H-EB
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 4
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 1149.4
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D2 2.0000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -2.00 dB
SFO1 300.1321009 MHz

F2 - Processing parameters
SI 16384
SF 300.1300078 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -1.000 ppm
F2 -300.13 Hz
PPHCP 0.55000 ppm/cm
HZCM 165.07150 Hz/cm

Current Data Parameters
NAME xuedongsnh
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061201
Time 15.02
INSTRUM av300
PROBHD 5 mm BBI 1H-EB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 0
SWH 18115.341 Hz
FIDRES 0.276427 Hz
AQ 1.0000436 sec
RG 35.0
DW 27.000 usec
DE 5.00 usec
TE 300.0 K
D1 2.0000000 sec
d11 0.0300000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PL1 -6.00 dB
SFO1 75.4752958 MHz

===== CHANNEL f2 =====
PULPROG zgpg30
NUC2 1H
PCPG2 80.00 usec
PL2 20.00 dB
PL12 17.50 dB
SFO2 300.1309004 MHz

F2 - Processing parameters
SI 131072
SF 75.4677490 MHz
WDW EM
SSB 0
LB 7.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 230.000 ppm
F1 16602.90 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPHCP 11.00000 ppm/cm
HZCM 830.14520 Hz/cm