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Supporting Information

Multicomponent One-Pot Procedure for the Synthesis of Free α -Chiral Amines from Aldehydes

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General: All non-aqueous reactions were run under argon with rigid exclusion of moisture from reagents and glassware using standard Schlenk techniques for manipulating air-sensitive compounds.¹ All glassware was stored in an oven and/or was flame-dried prior to use under an inert atmosphere of gas. Anhydrous solvents were obtained either by filtration through drying columns (Toluene and MeCN) or by distillation over sodium/benzophenone (Toluene). Analytical thin-layer chromatography (TLC) was performed on precoated, glass-backed silica gel plates. Visualization of the developed chromatogram was performed using UV absorbance, a ninhydrin solution or an aqueous potassium permanganate solution. Flash column chromatography was performed using 230-400 mesh silica gel and the indicated solvent system according to a standard technique.² Purification by preparative HPLC was performed on an apparatus equipped with a diode array UV detector, an auto sampler, and a reverse phase column. The eluent was a mixture of MeCN/H₂O under gradient conditions. Melting points are uncorrected. Infrared spectra were obtained on a FTIR equipped with a golden gate diamond ATR with ZnSe lenses and are reported in reciprocal centimeters (cm⁻¹).

¹ Shriver, D.F.; Drezdson, M. A. the manipulation of air-sensitive compounds; 2nd ed.; Wiley: New York, 1986.

² Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923–2925.

Nuclear magnetic resonance spectra (^1H , ^{13}C , ^{19}F , ^{31}P) were recorded on one of AV 300, AMX 300, AV 400 or ARX 400 spectrometer equipped with a BBO, a QNP or a DUAL probe. Chemical shifts for ^1H NMR spectra are recorded in parts per million using the residual solvent resonance as the internal standard (CD_2Cl_2 , δ 5.32 ppm or CD_3OD , δ 3.31 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, qn = quintet, sx = sextet, sp = septet, oc = octet, no = nonet, dc = decet, m = multiplet and br = broad). Coupling constants and integration values are reported in hertz. Chemical shifts for ^{13}C NMR spectra are recorded in parts per million from tetramethylsilane by calibrating the central peak of CD_2Cl_2 to 128.06 ppm and CD_3OD to 49.00 ppm. Chemical shifts for ^{31}P and ^{19}F NMR spectra are recorded in parts per million using external standards (phosphoric acid δ 0.0 ppm and trifluorotoluene δ -63.9 ppm). ^{13}C , ^{19}F and ^{31}P spectra were obtained with complete proton decoupling. Optical rotations were determined at 589 nm. Data are reported as follows: $[\alpha]_D^{25}$, concentration (c in g/100 mL), and solvent. Analytical High Performance Liquid Chromatography was performed on an analytical instrument equipped with a diode array UV detector. Data are reported as follows: (column type, eluent, flow rate: retention time (t_r)). Analytical Supercritical Fluid Chromatography was performed on an instrument equipped with a diode array UV detector. Data are reported as follows: column type, eluent, flow rate: retention time (t_r). Analytical gas chromatography was carried out on an instrument equipped with a splitless mode capillary injector and a flame ionization detector. Unless otherwise noted, the injector and detector temperatures were set to 250 °C and hydrogen was used as the carrier gas (63 psi).

Reagents: Neat diethylzinc, $(\text{CuOTf})_2 \cdot \text{toluene}$, neat dimethylzinc, aldehydes and all other chemical compounds were purchased. Unless otherwise stated, commercial reagents were used without purification. BozPHOS was synthesized according to a previously reported procedure.³

Diphenylphosphinoylamide was prepared by the following procedure: A

³ Boezio, A. A.; Pytkowicz, J.; Côté, A.; Charette, A. B. *J. Am. Chem. Soc.* **2003**, *125*, 14260–14261.

flame-dried 2-L, one-necked, round-bottomed flask, equipped with an egg-shaped magnetic stirring bar, a rubber septum and an internal thermocouple probe purged with argon. The flask is then charged with anhydrous dichloromethane (1 L) under argon. The septum is removed and acetone oxime (16.56 g, 227 mmol) is added quickly in one portion and the flask is sealed and purged again with argon. Freshly distilled triethylamine (31.6 mL, 227 mmol) is added via a syringe and the resulting colorless solution is cooled to -78°C (internal temperature) with a acetone/dry ice bath (Approximately 30 min are needed to reach this temperature). Freshly distilled colorless chlorodiphenylphosphine (42 mL, 227 mmol) is added dropwise (2 mL/min) via a syringe at such a rate that the internal temperature does not exceed -70°C . After the addition is complete, the resulting milky solution is stirred 15 min at -78°C under argon. The cooling bath is removed, and the solution is allowed to warm to room temperature over a period of 1.5 hr and stirred 1 hr at that temperature. The milky solution becomes clear. The solvent is removed under reduced pressure and the resulting off-white to yellow solid is dried under vacuum for 12 hr. The residue is redissolved in acetone (200 mL) and dried under reduced pressure. This last step can be repeated once again if the solid is gummy. The solid is removed from the flask and ground to a powder using a mortar and pestle. The powder is added in one portion to an Erlenmeyer flask containing a stirring solution of concentrated aqueous ammonium hydroxide (660 mL) and distilled water (330 mL). The mixture is usually heterogeneous and the precipitate is a white solid. Sometimes the solid added dissolves rapidly to form a clear yellow solution and, after few seconds, a white solid starts to precipitate. The heterogeneous mixture is stirred for 45 min and **1** is obtained as a white solid by filtration through a sintered glass funnel. The aqueous filtrate is extracted with dichloromethane (3 x 600 mL). The combined extracts are dried over Na_2SO_4 , filtered and evaporated under reduced pressure. The white solid obtained is combined with the white precipitate obtained above after filtration. The combined solids are dissolved in benzene (300 mL) and concentrated under reduced pressure. This step was repeated three times to remove traces of water to afford 46.31-47.87 g (94-97% yield)

General procedure to prepare racemic free α -chiral ammonium chloride.

Derivatives from arylaldehydes were synthesized by addition of Grignard reagents to trimethylsilylimines⁴ followed by cleavage of the silyl group under acidic conditions.

Derivatives from alkylaldehydes were synthesized by addition of Grignard reagents or a CuCN/R₂Zn mixture to the sulfinic acid adduct⁵ followed by acidic treatment in the case where a free amine was required. (The separation of both enantiomers is facilitated by the presence of the diphenylphosphinoyl group.)

(1S)-1-phenylpropan-1-ammonium chloride (2a). The general procedure was followed, with the exception that a 0.2M solution was used and Et₂Zn was added at 25 °C. Isolated yield 88%, 97% ee. GC analysis of the neutralized product on chiral stationary phase (β-Dex AD, 90 to 120 °C, 1 °C/min): (*R*)-**2a** *t_r* = 16.76 min, (*S*)-**2a** *t_r* = 16.96 min). All analytical data are in accordance with the literature.⁶ [α]_D²³ +17.8 ° (*c* 2.08, EtOH) compare to known value [α]_D +17.9 ° (*c* 2.1, EtOH).⁷

(1S)-1-(2-chlorophenyl)propan-1-ammonium chloride (2b). The general procedure was followed. A 0.1M solution was used and Et₂Zn was added at 0 °C. Isolated yield 54%, 96% ee. GC analysis of the

⁴ (a) Colvin, E. W.; McGarry, D. G. *J. Chem. Soc., Chem. Commun.* **1985**, 9, 539–540. (b) Ojima, I.; Habus, I.; Zhao, M.; Zucco, M.; Park, Y. H.; Ming, C.; Brigaud, T. *Tetrahedron* **1992**, 48, 6985–7012. (c) Flögel, O.; Reissig, H.-U. *Synlett* **2004**, 895–897. (d) Gosselin, F.; O'Shea P. D.; Roy, S.; Reamer, R. A.; Chen, C.-y.; Volante, R. P. *Org. Lett.* **2005**, 7, 355–358.

⁵ Côté, A.; Boezio, A. A.; Charette, A. *Proc. Natl Acad. Sci. USA* **2004**, 101, 5405–5410.

⁶ Cogan, D. A.; Liu, G.; Ellman, J. *Tetrahedron* **1999**, 55, 8883–8904.

⁷ La Manna, A. *et al. Farmaco Ed. Sci.* **1967**, 22, 1037–1053.

neutralized product on chiral stationary phase (β -Dex, 100 to 125 °C, 0.5 °C/min): (*R*)-**2b** t_r = 28.17 min, (*S*)-**2b** t_r = 28.60 min). mp 185.0–188.0 °C; R_f 0.60 (20% MeOH in CH_2Cl_2); $[\alpha]_D^{23}$ +19.6 ° (c 1.09, CD_3OD); ^1H NMR (400 MHz, CD_3OD) δ 7.59 (dd, J = 7.8, 1.5 Hz, 1H), 7.56 (dd, J = 7.8, 1.3 Hz, 1H), 4.78 (dd, J = 8.7, 6.5 Hz, 1H), 2.10 (dqn, J = 14.0, 7.0 Hz, 1H), 2.02 (dqn, J = 14.3, 7 Hz, 1H), 0.96 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, CD_3OD) δ 133.9, 133.2, 129.9, 129.6, 127.5, 126.7, 52.1, 26.6, 8.4; IR (neat) 2926, 2861, 1602, 1521, 1481, 1453, 1436, 1050, 1035, 762, 695 cm^{-1} ; LRMS (APCI+) $[\text{M}-\text{Cl}]^+$ found: 170.2 m/z .

(1S)-1-(2-methoxyphenyl)propan-1-ammonium chloride (2c). The

general procedure was followed. A 0.1M solution was used and Et_2Zn was added at 25 °C. Isolated yield 90%, 95% ee. GC analysis of the

neutralized product on chiral stationary phase (β -Dex, 100 to 120 °C, 0.1 °C/min): (*S*)-**2c** t_r = 46.15 min, (*R*)-**2c** t_r = 49.32 min. mp 140.0–141.0 °C; R_f 0.43 (20% MeOH in CH_2Cl_2); $[\alpha]_D^{23}$ +19.9 ° (c 1.22, MeOH); ^1H NMR (400 MHz, CD_3OD) δ 7.43 (td, J = 7.8, 1.7 Hz, 1H), 7.34 (dd, J = 7.5, 1.5 Hz, 1H), 7.13 (d, J = 8.2 Hz, 1H), 7.05 (td, J = 7.5, 0.8 Hz, 1H), 4.40 (dd, J = 8.2, 7.0 Hz, 1H), 2.11–1.99 (m, 2H), 0.92 (t, J = 7.4, 3H); ^{13}C NMR (100 MHz, CD_3OD) δ 156.9, 129.9, 127.9, 123.5, 120.3, 110.7, 54.3, 52.7, 25.3, 9.0; IR (neat) 2937, 2857, 2051, 1605, 1518, 1498, 1454, , 1245, , 1024, 759 cm^{-1} ; LRMS (APCI+) $[\text{M}-\text{Cl}]^+$ found: 166.2 m/z .

(1S)-1-(2-methylphenyl)propan-1-ammonium chloride (2d). The general procedure was followed, with the exception that a 0.2M solution was used and Et₂Zn was added at 25 °C. Isolated yield 68%, 96% ee. GC analysis of the neutralized product on chiral stationary phase (β-Dex, 80 to 100 °C, 0.1 °C/min): (*S*)-**2d** *t*_r = 64.83 min, (*R*)-**2d** *t*_r = 66.99 min). mp 221.0–222.0 °C; *R*_f 0.40 (20% MeOH in CH₂Cl₂); [α]_D²³ +23.2 ° (*c* 0.93, CD₃OD); ¹H NMR (400 MHz, CD₃OD) δ 7.44 (d, *J* = 7.7 Hz, 1H), 7.38–7.31 (m, 2H), 7.30 (d, *J* = 1.2 Hz, 1H), 4.52 (dd, *J* = 8.8, 6.2 Hz, 1H), 2.44 (s, 3H), 2.13–1.91 (m, 2H), 0.93 (t, *J* = 7.6 Hz, 3H); ¹³C NMR (100 MHz, CD₃OD) δ 136.0, 134.8, 130.4, 128.2, 126.4, 124.4, 51.5, 27.1, 17.7, 8.5; IR (neat) 2876, 2015, 1596, 1516, 1462, 1379, 1033, 759, 727 cm⁻¹; LRMS (APCI+) [*M*-Cl]⁺ found: 150.3 *m/z*.

(1S)-1-(1-naphthyl)propan-1-ammonium chloride (2e). The general procedure was followed, with the exception that a 0.2M solution was used and Et₂Zn was added at 25 °C. Isolated yield 59%, 97% ee. HPLC analysis of the diphenylphosphinoylated product on chiral stationary phase (Chiralpak AD, 90:10 Hexane: *i*-PrOH, 1mL/min): (*R*)-**2e** *t*_r = 14.75 min, (*S*)-**2e** *t*_r = 24.86 min). mp 235.0–236.0 °C; *R*_f 0.40 (20% MeOH in CH₂Cl₂); [α]_D²³ +17.2 ° (*c* 1.29, CD₃OD); ¹H

NMR (400 MHz, CD₃OD) δ 8.21 (d, J = 8.6 Hz, 1H), 8.00 (d, J = 3.4 Hz, 1H), 7.98 (d, J = 3.4, 1H), 7.71-7.57 (m, 4H), 5.22 (t, J = 7.4 Hz, 1H), 2.19 (dc, J = 7.4 Hz, 2H), 0.98 (t, J = 7.4, 3H); ¹³C NMR (100 MHz, CD₃OD) δ 133.7, 132.7, 130.6, 128.9, 128.5, 126.5, 125.6, 124.7, 122.7, 121.5, 50.7, 27.5, 8.7; IR (neat) 2916, 2844, 1600, 1522, 1454, 800, 777 cm⁻¹; LRMS (APCI+) [M-Cl]⁺ found: 186.2 m/z ; Elemental analysis calcd for C₁₃H₁₆ClN: C, 70.42; H, 7.27; N, 6.32, found: C, 70.14; H, 7.02; N, 6.35.

(1S)-1-(3-nitrophenyl)propan-1-ammonium chloride (2f). The general procedure was followed. A 0.1M solution was used and Et₂Zn was added at 0 °C. Isolated yield 79% yield, 97% ee. GC analysis of the neutralized product on chiral stationary phase (β-Dex, 130 to 160 °C, 0.5 °C/min): (*R*)-**2f** t_r = 38.91 min, (*S*)-**2f** t_r = 39.34 min). mp 232.0–231.0 °C; R_f 0.54 (20% MeOH in CH₂Cl₂); $[\alpha]_D^{23}$ +19.5 ° (c 1.08, MeOH); ¹H NMR (400 MHz, CD₃OD) δ 8.42 (t, J = 1.9 Hz, 1H), 8.34 (ddd, J = 8.2, 2.2, 1.0 Hz, 1H), 7.90 (dt, J = 7.8, 1.0 Hz, 1H), 7.77 (t, J = 8.0 Hz, 1H); ¹³C NMR (100 MHz, CD₃OD) δ 148.3, 138.5, 133.1, 130.0, 123.3, 121.6, 55.6, 26.9, 8.6; IR (neat) 2858, 2621, 2038, 1606, 1517, 1482, 1351, 1314, 1106, 818, 803, 739, 691, 679 cm⁻¹; LRMS (APCI+) [M-Cl]⁺ found: 181.2 m/z ; Elemental analysis calcd for C₉H₁₃ClN₂O₂: C, 49.89; H, 6.05; N, 12.93, found: C, 49.81; H, 5.87; N, 12.72.

(1S)-1-(2-furyl)propan-1-ammonium chloride (2g). The specific procedure describes in the experimental section was followed. Isolated yield 65%, 93% ee, (highly hygroscopic). SFC analysis of the diphenylphosphinoylated product on chiral stationary phase (Chiralpak AS, 15% i-PrOH, 2mL/min, 200 bar, 30°C): (*R*)-**2g** t_r = 15.89 min, (*S*)-**2g** t_r = 20.10 min). mp 137.0–140.0 °C; R_f 0.43 (20% MeOH in CH_2Cl_2); $[\alpha]_D^{23} +13.4^\circ$ (c 1.43, CD_3OD); ^1H NMR (400 MHz, CD_3OD) δ 7.63 (dd, J = 1.9, 0.8 Hz, 1H), 6.57 (ddd, J = 3.3, 0.8, 0.3 Hz, 1H), 6.50 (dd, J = 3.4, 1.8 Hz, 1H), 4.35 (dd, J = 9.1, 5.8 Hz, 1H), 2.13–1.94 (m, 2H), 0.96 (t, J = 7.5 Hz, 3H); ^{13}C NMR (100 MHz, CD_3OD) δ 148.8, 143.2, 110, 109, 49.6, 24.5, 8.6; IR (neat) 3394, 2973, 1607, 1506, 1366, 1244, 1153, 1012, 929, 747, 600 cm^{-1} ; Elemental analysis calcd for $\text{C}_7\text{H}_{12}\text{ClNO}$: C, 52.02; H, 7.48; N, 8.67 found: C, 51.47; H, 7.85; N, 8.54.

(1S)-1-ethyl-2-methylpropylammonium chloride (2h). The general procedure was followed, with the exception that a 0.2M solution was used and Et_2Zn was added at 25 °C. Isolated yield 62%, 93% ee. HPLC analysis of the diphenylphosphinoylated product on chiral stationary phase (Chiralpak AD-H,

90:10 Hexane: *i*-PrOH, 1mL/min): (*S*)-**2h** t_r = 13.25 min, (*R*)-**2h** t_r = 14.87 min). mp 210.0–211.0 °C; R_f 0.40 (20% MeOH in CH₂Cl₂); $[\alpha]_D^{23}$ -3.8 ° (*c* 1.03, CD₃OD)⁸; ¹H NMR (400 MHz, CD₃OD) δ dt, J = 7.8, 5.1 Hz, 1H), 2.05–1.93 (m, 1H), 1.81–1.69 (m, 1H), 1.62 (sp, J = 7.6 Hz, 1H), 1.04 (t, J = 7.2 Hz, 3H), 1.04 (d, J = 3.6 Hz, 3H), 1.02 (d, J = 4.0 Hz, 3H); ¹³C NMR (75 MHz, CD₃OD) δ 58.9, 29.8, 22.6, 17.4, 17.0, 9.0; IR (neat) 2965, 2881, 2024, 1603, 1515, 1462, 1397 cm⁻¹; LRMS (APCI+) [M-Cl]⁺ found: 102.3 m/z .

(1S)-1-ethyl-3-phenylpropylammonium chloride (2i). The general procedure was followed. A 0.1M solution was used and Et₂Zn was added at 0 °C. Isolated yield 59%, 90% ee. HPLC analysis of the

diphenylphosphinoylated product on chiral stationary phase (Chiralpak AD-H, 80:20 Hexane: *i*-PrOH, 1mL/min): (*R*)-**2i** t_r = 9.31 min, (*S*)-**2i** t_r = 13.01 min). mp 185.0–186.0 °C; R_f 0.38 (20% MeOH in CH₂Cl₂); $[\alpha]_D^{23}$ -17.8 ° (*c* 1.58, MeOH); ¹H NMR (400 MHz, CD₃OD) δ 7.32 (t, J = 7.2 Hz, 2H), 7.25 (d, J = 7.4 Hz, 2H), 7.22 (t, J = 7.1 Hz, 1H), 3.15 (qn, J = 6.4 Hz, 1H), 2.74 (ddd, J = 9.4, 6.4, 2.1 Hz, 2H), 2.03–1.85 (m, 2H), 1.85–1.75 (m, 1H), 1.70 (oc, J = 7.2 Hz, 1H), 1.04 (t, J = 7.6 Hz, 3H); ¹³C NMR (75 MHz, CD₃OD) δ 140.9, 128.7, 128.3, 126.4, 52.9, 34.3, 31.3, 25.3, 8.6; IR (neat) 2882, 1619, 1602, 1514, 1452, 1393, 764, 742, 699 cm⁻¹; LRMS (APCI+) [M-Cl]⁺ found: 164.3 m/z .

⁸ Cogan, D. A.; Lui, G.; Ellman, J. *Tetrahedron* **1999**, 55, 8883–8904. Literature value: $[\alpha]_D^{23}$ -12.0 (*c* 4.0, MeOH).

(1S)-1-phenylethanammonium chloride (2j). The general procedure for

the addition of dimethylzinc was followed. For the reaction over 48 h:

32% yield, 92% ee. For the reaction over 96 h: 41% yield, 90% ee. GC

analysis of the neutralized product on chiral stationary phase (β -Dex, 90 to 120 °C, 0.5

°C/min): (*R*)-**2j** t_r = 12.86 min, (*S*)-**2j** t_r = 13.14 min). All analytical data are in

accordance with the literature.⁹ $[\alpha]_D^{23}$ -3.9 ° (c 1.95, EtOH) compare to the known value for the other enantiomer $[\alpha]_D^{23}$ +6.7 ° (c 2.0, EtOH).¹⁰

⁹ Boezio, A. A.; Solberghe, G.; Lauzon, C.; Charette, A. B. *J. Org. Chem.* **2003**, 68, 3241–3245.

¹⁰ Smith, H. E.; Gordon, A. W.; Bridges, A. F. *J. Org. Chem.* **1974**, 39, 2309–2311.


```

Current Data Parameters
NAME      AC-5-189
EXPNO     3
PROCNO    1

F2 - Acquisition Parameters
Date_     20050521
Time      21.19
INSTRUM   spect
PROBHD    5 mm QNP 1H/1
PULPROG   zg30
TD         45000
SOLVENT   CDCl2
NS         32
DS         2
SWH        3858.023 Hz
FIDRES     0.08573 Hz
AQ          5.8320498 sec
RG          28.5
DW         129.600 usec
DE         6.00 usec
TE         292.2 K
D1          1.00000000 sec
MCREST     0.00000000 sec
MCWRK      0.01500000 sec

===== CHANNEL f1 =====
NUC1       1H
P1          8.50 usec
PL1         6.00 dB
SFO1       300.1316821 MHz

F2 - Processing parameters
SI          65536
SF          300.1300051 MHz
WDW         no
SSB         0
LB          0.00 Hz
GB          0
PC          1.00

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1, ^1H NMR (CD_2Cl_2 , 400 MHz)

```

Current Data Parameters
NAME      AC-S-189
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20050521
Time      21.14
INSTRUM   spect
PROBHD    5 mm QNP 1H/1
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         32
DS         2
SWH        75187.969 Hz
FIDRES     1.147277 Hz
AQ         0.4358644 sec
RG         2298.8
DW         6.650 usec
DE         6.00 usec
TE         298.2 K
D1         1.00000000 sec

===== CHANNEL f1 =====
NUC1       19F
P1         7.30 usec
PL1        0.00 dB
PL12       10.00 dB
SF01       282.3875367 MHz

F2 - Processing parameters
SI         131072
SF         282.4044810 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00

```

-79.102

p]

-120

-100

-80

-60

-40

-20

0

20

40

2c, ^1H NMR (CD_3OD , 400 MHz)

2d, ^1H NMR (CD_3OD , 100 MHz)

Current Data Parameters
NAME AC-5-59-prep-3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050623
Time 2.16
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 65
DSH 4504.504 Hz
SFO1 400.1380000 MHz
FIDRES 0.137467 Hz
AQ 3.6372981 sec
RG 1024
DE 111.000 usec
TE 298.0 K
D1 1.00000000 sec
P1 8.00 usec
SFO1 400.1380000 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 32768
SF 400.1359660 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

2e, ^1H NMR (CD_3OD , 400 MHz)

2e, ^{13}C NMR (CD_3OD , 100 MHz)

Current Data Parameters
NAME AC-5-175-Prep
PROCNO 1
F2 - Acquisition Parameters
Date 20050221
Time 6.09
INSTRUM av400
PROBHD 5 mm PABBO
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 8192
DS 2
SWH 25062.656 Hz
FIDRES 0.489305 Hz
AQ 0.000000 sec
RG 32768
DW 19.950 usec
DE 6.00 usec
TE 300.2 K
D1 1.50000000 sec
d11 0.03000000 sec
DELTA 1.33000000 sec
DELTAT 0.00000000 sec
MORPH 0.01000000 sec
===== CHANNEL F1 =====
NUC1 ¹³C
P1 7.90 usec
PL1 -3.00 dB
SFO1 100.6242690 MHz
===== CHANNEL F2 =====
PULPROG waltz16
NUC2 ¹H
PCPD2 96.00 usec
PL2 -3.00 dB
PL12 16.00 dB
PL13 16.00 dB
SFO2 400.1315000 MHz
F2 - Processing parameters
SI 32768
SF 100.6128074 MHz
WDW no
SSB 0.00 Hz
GB 0
PC 1.40

2g, ¹H NMR (CD₃OD, 400 MHz)

Current Data Parameters
NAME AC-5-165-prep
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050531
Time 16.17

INSTRUM av400
PROBHD 5 mm EABBO BB-
PULPROG zg30
TD 49152
SOLVENT CDCl₃
NS 12
DS 2

SWH 5995.204 Hz
FIDRES 0.121973 Hz
AQ 4.0993266 sec
RG 256
DW 83.400 usec
DE 6.00 usec
TE 292.3 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MORPK 0.01500000 sec

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

NUC1 ¹H
P1 10.00 usec
PL1 2.00 dB
SFO1 400.1326000 MHz

F2 - Processing parameters

SI 65536

SF 400.1300000 MHz

WDW no

SSB 0

LB 0.00 Hz

GB 0

PC 1.00

Current Data Parameters
NAME AC-5-65-prep-3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050623
Time 2.09
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 54
DSH 4504.504 Hz
SFO1 400.138000 MHz
FIDRES 0.137467 Hz
AQ 3.6372981 sec
RG 111.000 usec
DE 158.57 usec
TE 298.0 K
D1 1.00000000 sec
P1 8.00 usec
SFO1 400.138000 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 32768
SF 400.1359660 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

2h, ¹H NMR (CD₃OD, 400 MHz)

2i, ¹H NMR (CD₃OD, 400 MHz)

Current Data Parameters
NAME AC-5-173-prep
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050606
Time 0.02
INSTRUM av400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 49352
SOLVENT CDCl₃
NS 501
DS 2
SWH 5995.204 Hz
FIDRES 0.121973 Hz
AQ 4.0993266 sec
RG 256
DW 83.400 usec
DE 6.00 usec
TE 292.3 K
L1 1.0000000 sec
MCREST 0.0000000 sec
MCWRK 0.01500000 sec
===== CHANNEL f1 =====
NUC1 1H
P1 10.00 usec
PL1 -3.00 dB
SFO1 400.1326000 MHz
F2 - Processing parameters
SI 65536
SF 400.1300000 MHz
WDW no
SSB 0
GB 0.00 Hz
PC 1.00