Synthesis of 1,2-Dihydropyridines Using Vinyloxiranes as Masked Dienolates in Imino-Aldol-Reactions.

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1. General

Reactions were generally carried out under nitrogen atmosphere in an appropriate round bottom flask with magnetic stirring. Tetrahydrofuran (THF) and toluene were distilled under N₂-atmosphere from Na/benzophenone immediately prior to use.

Analytical Thin layer chromatography was performed with SilicycleTM 0.25 mm plastic backed silica gel TLC plates. Visualization was accomplished with 254 nm UV light and potassium permanganate solution (unless otherwise noted). Purification of reaction products was generally done by flash chromatography with SilicycleTM Ultra-Pure 230-400 mesh silica gel. Melting points were taken on a Fisher-Johns melting point apparatus and are uncorrected. IR spectra were obtained using a Perkin-Elmer Spectrum 1000 FT-IR spectrometer. ¹H and ¹³C NMR spectra were recorded on a Mercury 300 (¹H: 300 MHz, ¹³C: 75 MHz) or a Mercury 400 (¹H: 400 MHz, ¹³C: 100 MHz) spectrometer. Spectral features are tabulated in the following order: chemical shift (δ in ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = complex multiplet), coupling constant (*J* in Hz), number of protons. High resolution mass spectra were obtained from a Micromass 70S-250 mass spectrometer (EI) or an ABI/Sciex Qstar spectrometer (ESI). Sc(OTf)₃ was purchased from Strem Chemicals, Inc.; BF₃·OEt₂, Iodobenzene diacetate (BAIB) and TEMPO were purchased from Sigma-Aldrich Canada Ltd. IUPAC names of the compounds described in the experimental section were determined with the program ACD/I-LAB 8.0.

The characterisation data and spectra for several compounds (aldimines) have been reported previously by us (**2b**, **2d**, **2h**, **2k**, **2i**).^[1]

^[1a] Lautens, M.; Tayama, E.; Nguyen, D.; *Org. Lett.* **2004**, 6, 345-347; ^[1b] Lautens, M.; Tayama, E.; Nguyen, D.; *Tetrahedron Lett.* **2004**, 45, 5131-5133; ^[1c] Lautens, M.; Tayama, E.; Herse, C.; *J. Am. Chem. Soc.* **2005**, 127, 72-73.

2. Synthesis of the Vinyloxiranes

The substituted vinyloxiranes were synthesized by using a one-pot two-step procedure (see **GP II**). For this the epoxy alcohols were first oxidized by using a procedure described by Margarita and Piancatelli² and the formed epoxy aldehyde was subsequently reacted with the corresponding Wittig reagent. With this method the vinyloxiranes were synthesized in yields >70 % and an E:Z-ratio >15:1.

General Procedure for the synthesis of the vinyloxiranes (GP I):

TEMPO (10 mol %) was added to a solution of the corresponding epoxy alcohol (1.0 equiv) in dry CH_2Cl_2 (0.4 M-solution). Afterwards BAIB (1.2 equiv.) was added in small portions to the reaction mixture. The reaction mixture was stirred under a N_2 -atmosphere at room temperature (2-4 h) until a complete conversion of the epoxy alcohol could be detected. Afterwards the reaction mixture was cooled to 0 °C and neutralized through the addition of NEt_3 (2.4 equiv.). A solution of the corresponding Wittig-reagent in CH_2Cl_2 was added dropwise to the reaction mixture. The reaction mixture was stirred for additional 3.5 h at 0 °C. The solvent was evaporated under reduced pressure and the residue was dissolved in diethyl ether. The solution was stored for several hours at 0 °C and the precipitation was removed via filtration through silica gel. The solvent was removed under a reduced pressure and the residue was purified via column chromatography on silica.

Ethyl (2E)-3-(2-methyloxiran-2-yl)acrylate (1a): (2-Methyl-oxiranyl)-methanol (1.76 g, 20 mmol) was dissolved in 50 ml dry CH_2Cl_2 . Oxidation was performed by addition of TEMPO (312 mg, 2.0 mmol) and BAIB (7.08

g, 22 mmol) by using the General Procedure from above (**GP I**). Afterwards NEt_3 (5.54 ml, 40 mmol) and MeOOCCHPh_3 (7.5 g, 22 mmol, dissolved in 25 ml dry CH_2Cl_2) were added according to **GP II**. After work up and purification by column chromatography over silica 2.44 g (75 %) of **1a** were obtained as colorless oil. R_f = 0.3 (hexane:diethylether 4:1). IR (neat): 2930, 1728, 1306, 1167, 979, 721 cm^{-1} . ^1H NMR (300 MHz, CDCl_3): δ 6.70 (d, 3J = 15.8 Hz, 1 H), 6.05 (d, 3J = 15.8 Hz, 1 H), 4.19 (q, 3J = 7.2 Hz, 2 H), 2.89 (d, J_{AB} = 5.3 Hz, 1 H), 2.79 (d, J_{AB} = 5.3 Hz, 1 H), 1.50 (s, 3 H), 1.28 (t, 3J = 7.2 Hz, 3 H). ^{13}C NMR (300 MHz, CDCl_3): δ

^[2] De Mico, A.; Mararita, R.; Parlanti, L.; Vescovi, A.; Piancatelli, G.; *J. Org. Chem.* **1997**, 62, 6974-6977.

165.9, 148.3, 122.7, 60.6, 55.9, 54.7, 19.0, 14.2. HRMS (EI, $[M+H]^+$): calcd. for $C_8H_{13}O_3$, 157.0865; found: 157.0867.

Methyl (2E)-3-(2-phenyloxiran-2-yl)acrylate (1b): (2-Phenyl-oxiranyl)-methanol (290 mg, 1.93 mmol) was dissolved in 10 ml dry CH_2Cl_2 .

Oxidation was performed by addition of TEMPO (30 mg, 0.19 mmol) and BAIB (684 mg, 2.12 mmol) by using the General Procedure from above (**GP I**). Afterwards NEt_3 (0.53 ml, 3.86 mmol) and $MeOOCCHPh_3$ (806 mg, 2.41 mmol, dissolved in 5 ml CH_2Cl_2) were added according to **GPI**. After work up and purification by column chromatography over silica 293 mg (74 %) of **1b** were obtained as colorless oil. Using the General Procedure from above (**GP I**). R_f = 0.31 (hexane:diethylether 4:1). IR (neat): 3011, 2723, 1732, 1657, 1312, 1266, 1167, 977, 913, 738 cm^{-1} . 1H NMR (400 MHz, $CDCl_3$): δ 7.36-7.40 (m, 5 H), 7.11 (d, 3J = 15.6 Hz, 1 H), 5.97 (d, 3J = 15.6 Hz, 1 H), 3.74 (s, 3 H), 3.14 (d, J_{AB} = 5.7 Hz, 1 H), 3.12 (d, J_{AB} = 5.7 Hz, 1 H). ^{13}C NMR (400 MHz, $CDCl_3$): δ 166.3, 146.7, 136.4, 128.5, 128.4, 126.9, 123.6, 59.1, 57.4, 51.7. HRMS (EI, $[M-H]^+$): calcd. for $C_{12}H_{11}O_3$, 203.0708; found: 203.0713.

3. Synthesis of the Aldimines³

General Procedure for the synthesis of the aldimines (GP II):

The corresponding aldehyde (1.0 equiv.) and amine (1.0 equiv. aminodiphenylmethane or 1.0 equiv. of the aniline derivative) were dissolved in toluene (0.5 M-solution). Molecular sieves (5 Å) was added and the reaction mixture was stirred for 10 h at room temperature. The molecular sieves were removed by filtration and the solvent was removed under reduced pressure. The residue was either purified by crystallization or Kugelrohr-distillation.

³ Yields are not optimized.

***N*-[(1*E*)-(4-fluorophenyl)methylene]-1,1-diphenylmethanamine (2a):**

Using the General Procedure from above (**GP II**). Colorless crystals; purified by recrystallization from diethylether; yield 71 %. M. p. = 75 °C.

IR (film): 3061, 2852, 1644, 1602, 1377, 1024, 831, 699. ¹H NMR (300 MHz, CDCl₃): δ 8.38 (s, CH=N, 1 H), 7.81-7.85 (m, 2 H), 7.38-7.41 (m, 4 H), 7.29-7.34 (m, 4 H), 7.20-7.25 (m, 2 H), 7.06-7.12 (m, ³J = 8.4 Hz, 2 H), 5.58 (s, CHPh₂, 1 H). ¹³C NMR (100 MHz, CDCl₃): δ 164.3 (d, ¹J_{CF} = 250.7 Hz, 1 C), 159.3, 143.8, 132.6 (d, ⁴J_{CF} = 3.1 Hz, 1 C), 130.3 (d, ³J_{CF} = 8.8 Hz, 1 C), 128.4, 128.0, 127.6, 127.0, 115.6 (d, ²J_{CF} = 21.8 Hz, 1 C), 77.4. HRMS (EI, [M]⁺): calcd. for C₂₀H₁₆FN, 289.1267; found: 289.1267.

***N*-[(1*E*)-(4-bromophenyl)methylene]-1,1-diphenylmethanamine (2c):**

Using the General Procedure from above (**GP II**). Colorless crystals; purified by recrystallization from diethylether; yield 70 %. M. p. = 101 °C.

IR (film): 3060, 2849, 1641, 1589, 1452, 1010, 815. ¹H NMR (300 MHz, CDCl₃): δ 8.36 (s, CH=N, 1 H), 7.69-7.72 (m, ³J = 8.5 Hz, 2 H), 7.53-7.55 (m, ³J = 8.5 Hz, 2 H), 7.29-7.4 (m, 7 H), 7.21-7.26 (m, 2 H), 5.59 (s, CHPh₂, 1 H). ¹³C NMR (100 MHz, CDCl₃): δ 159.5, 143.6, 135.2, 131.8, 129.9, 128.5, 127.6, 127.1, 125.2, 77.9. HRMS (EI, [M]⁺): calcd. for C₂₀H₁₆BrN, 349.0466; found: 349.0459.

***N*-[(1*E*)-(2,4-dichlorophenyl)methylene]-1,1-diphenylmethanamine (2f).**

Using the General Procedure from above (**GP II**). Colorless crystals; purified by recrystallization from diethylether; yield 73 %. M. p. = 104 °C. IR (film): 3085, 2884, 1634, 1584, 1494, 1484, 1051, 831, 700 cm⁻¹. ¹H NMR (300 MHz, CDCl₃): δ 8.81 (s, CH=N, 1 H), 8.19 (d, ³J =

8.5 Hz, 1 H), 7.21-7.40 (m, 12 H), 5.65 (s, CHPh₂, 1 H). ¹³C NMR (100 MHz, CDCl₃): δ 156.4, 143.5, 137.1, 135.8, 131.8, 129.7, 129.5, 128.5, 127.6, 127.5, 127.1, 78.3. HRMS (EI, [M]⁺): calcd. for C₂₀H₁₅Cl₂N, 339.0582; found: 339.0578.

1,1-Diphenyl-*N*-[(1*E*)-[2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl]methylene]methanamine (2g).

Using the General Procedure from above (**GP II**). Pale yellow crystals; purified by recrystallization from diethylether; yield 70 %. M. p. = 80 °C. IR (nujol): 1639, 1554, 1139, 857, 657 cm⁻¹. ¹H NMR (300 MHz, CDCl₃): δ 9.05 (s, CH=N, 1 H), 8.20 (d, ³J = 7.7 Hz, 1 H), 7.82 (dd, ³J = 7.4 Hz, ⁴J = 1.2 Hz, 1 H), 7.23-7.49 (m, 12

H), 5.76 (s, CHPh₂, 1 H), 1.32 (s, 12 H). ¹³C NMR (100 MHz, CDCl₃): δ 162.9, 143.6, 141.8, 135.4, 130.8, 129.5, 128.3, 128.1, 126.9, 126.8, 83.8, 24.9. HRMS (EI, [M]⁺): calcd. for C₂₆H₂₈BNO₂, 397.2213; found: 397.2212.

2-Bromo-4-methoxy-N-[(1E)-phenylmethylene]aniline (2j): Using the General Procedure from above (**GP II**). Pale brown crystals; purified by recrystallization from diethylether; yield 68 %. M. p. = 69 °C. IR (film): 3060, 2834, 1625, 1592, 1483, 1285, 1032, 859, 690 cm⁻¹. ¹H NMR (300 MHz, CDCl₃): δ 8.37 (s, CH=N, 1 H), 7.91-7.95 (m, 2 H), 7.45-7.50 (m, 3 H), 7.20 (d, ⁴J = 2.7 Hz, 1 H), 7.02 (d, ³J = 8.7 Hz, 1 H), 6.87 (dd, ³J = 8.7 Hz, ⁴J = 2.7 Hz, 1 H), 3.81 (s, OMe, 3 H). ¹³C NMR (100 MHz, CDCl₃): δ 160.2, 158.1, 143.6, 136.1, 131.5, 128.9, 128.8, 119.7, 119.6, 118.1, 114.2, 55.7. HRMS (EI, [M]⁺): calcd. for C₁₄H₁₂BrNO, 289.0102; found: 289.0099.

N-[(1E)-2-furylmethylene]-1,1-diphenylmethanamine (2l): Using the General Procedure from above (**GP II**). Pale brown crystals; purified by recrystallization from diethylether; yield 68 %. M. p. = 105-106 °C. IR (neat): 3023, 2919, 1645, 1482, 913. ¹H NMR (300 MHz, CDCl₃): δ 8.19 (s, CH=N, 1 H), 7.52 (d, ³J = 1.7 Hz, 1 H), 7.29-7.38 (m, 8 H), 7.19-7.25 (m, 2 H), 6.79 (d, ³J = 3.5 Hz, 1 H), 6.46 (dd, ³J = 3.5 Hz, ³J = 1.7 Hz, 1 H), 5.58 (s, CHPh₂, 1 H). ¹³C NMR (100 MHz, CDCl₃): δ 151.7, 149.7, 144.9, 143.3, 128.4, 127.8, 127.0, 114.5, 111.6, 78.0. HRMS (EI, [M+H]⁺): calcd. for C₁₈H₁₅NO, 261.1154; found: 261.1150.

N-[(1E)-phenylmethylene]-2-(trifluoromethyl)aniline (2n): Using the General Procedure from above (**GP II**). Pale yellow crystals; purified by Kugelrohr-distillation (120-130 °C, 0.5 mm Hg); yield 49 %. IR (nujol): 2723, 1636, 1599, 1580, 1264, 758, 690. ¹H NMR (300 MHz, CDCl₃): δ 8.37 (s, CH=N, 1 H), 7.92 (m, 2 H), 7.68 (d, ³J = 7.6 Hz, 1 H), 7.45-7.57 (m, 4 H), 7.29 (d, ³J = 7.6 Hz, 1 H), 7.04 (d, ³J = 7.6 Hz, 1 H). ¹³C NMR (100 MHz, CDCl₃): δ 161.5, 150.8, 135.9, 132.9, 131.9, 129.2, 128.8, 126.3 (q, ³J_{CF} = 5.3 Hz, 1 C), 125.0, 124.0 (q, ¹J_{CF} = 273 Hz, 1 C), 123.1 (q, ²J_{CF} = 30.1 Hz, 1 C), 119.4. HRMS (EI, [M]⁺): calcd. for C₁₄H₁₀F₃N, 249.0765; found: 249.0758.

4. Synthesis of the 1,2-Dihydropyridines

Table 1. Solvent-screening for the reaction of vinyloxirane **1a** with aldimine **2a**

Entry ^a	Solvent	temperature	Time (h)	Conv 2a (%) ^b	Yield (%) ^c
1	CH ₂ Cl ₂	0 °C - 50 °C	4	35	n. d.
2	toluene	0 °C - rt	5	n. d.	36
3	toluene	0 °C - 50 °C	3	65	38
4	THF	0 °C - 50 °C	2.5	70	61
5	THF/H ₂ O	0 °C - 50 °C	2	no reaction	-----
6	CH ₃ OH	0 °C - 50 °C	2	no reaction	-----

^a All reactions were performed by using 0.75 mmol of **1a** and 0.5 mmol of **2b**. ^b Determined via ¹H NMR-spectroscopy from the crude product. ^c Isolated yields

General Procedure for the synthesis of 1,2-Dihydropyridines (GP III):

The vinyloxirane (0.75 mmol, 1.5 equiv.) and the corresponding aldimine (0.5 mmol, 1.0 equiv.) were dissolved in dry THF (1.0 ml). Molecular sieves (5 Å) were added and the solution was cooled to 0 °C. Sc(OTf)₃ (0.075 mmol, 37 mg, 15 mol %) was added under a N₂-atmosphere. The reaction mixture was allowed to warm to room temperature and was then heated to 50 °C. After of 2-3 h no further conversion of the starting materials could be observed. The reaction mixture was diluted with CH₂Cl₂ (10 ml) and the molecular sieves were filtered off. The filtrate was washed with a saturated NaHCO₃-solution (5 ml). The organic layer was dried over Na₂SO₄, filtered and the solvent was evaporated under reduced pressure. Purification of the residue by column chromatography on silica gel gave the corresponding 1,2-dihydropyridine.

Ethyl 1-(diphenylmethyl)-2-(4-fluorophenyl)-5-methyl-1,2-dihydropyridine-3-carboxylate (4a): Using the General Procedure from above (**GP III**). Yellow crystals; purified by column chromatography over silica gel; 130 mg (61 % yield). M. p. = 142 °C. R_f = 0.46 (hexane:diethylether 4:1). IR (Nujol): 2889, 1683, 1462, 1377, 1122, 1077, 739 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 7.29-7.43 (m, 5 H), 7.23-7.29 (m, 5 H), 7.12 (s, 1 H), 1.18 (t, 3J = 7.2 Hz, 3 H), 6.99 (m, 3J = 8.8 Hz, 2 H), 6.84-6.87 (m, 2 H), 6.09 (s, 1 H), 5.56 (s, 1 H), 5.54 (s, 1 H), 3.99-4.14 (m, 2 H), 1.68 (s, 3 H). ^{13}C NMR (300 MHz, CDCl_3): δ 166.3, 162.5 (d, $^1J_{\text{C,F}}$ = 245.3 Hz, 1 C), 140.0, 139.5 (d, $^4J_{\text{C,F}}$ = 3.1 Hz, 1 C), 139.2, 137.5, 136.9, 129.5, 128.8 (d, $^3J_{\text{C,F}}$ = 7.8 Hz, 1 C), 128.7, 128.6, 127.89, 127.86, 127.8, 115.1 (d, $^2J_{\text{C,F}}$ = 21.3 Hz, 1 C), 111.8, 100.6, 69.2, 60.1, 59.8, 17.1, 14.3. HRMS (EI, $[\text{M}]^+$): calcd. for $\text{C}_{28}\text{H}_{26}\text{FNO}_2$, 427.1948; found: 427.1947.

Ethyl 2-(4-chlorophenyl)-1-(diphenylmethyl)-5-methyl-1,2-dihydropyridine-3-carboxylate (4b): Using the General Procedure from above (**GP III**). Yellow oil; purified by column chromatography on silica gel; 134 mg (60 % yield). R_f = (hexane:diethylether 4:1). IR (neat): 3018, 1680, 1523, 1488, 1216, 1091, 1015, 760, 669 cm^{-1} . ^1H NMR (300 MHz, CDCl_3): δ 7.23-7.39 (m, 12 H), 7.12 (d, 4J = 1.1 Hz, 1 H), 6.87-6.91 (m, 2 H), 6.10 (d, 4J = 1.1 Hz, 1 H), 5.55 (s, 2 H), 3.99-4.15 (m, 2 H), 1.68 (s, 3 H), 1.19 (t, 3J = 7.2 Hz, 3 H). ^{13}C NMR (300 MHz, CDCl_3): δ 166.3, 141.9, 139.9, 139.1, 137.6, 136.9, 133.4, 129.5, 128.7, 128.6, 128.5, 127.9, 127.84, 127.81, 111.5, 100.8, 69.4, 60.1, 59.8, 17.1, 14.3. HRMS (EI, $[\text{M}]^+$): calcd. for $\text{C}_{28}\text{H}_{26}\text{ClNO}_2$, 443.1652; found: 443.1661.

Ethyl 2-(4-bromophenyl)-1-(diphenylmethyl)-5-methyl-1,2-dihydropyridine-3-carboxylate (4c): Using the General Procedure from above (**GP III**). Yellow oil; purified by column chromatography on silica gel; 153 mg (62 % yield). R_f = 0.45 (hexane:diethylether 4:1). IR (neat): 3027, 2976, 1686, 1526, 1300, 1190, 1011, 966, 700 cm^{-1} . ^1H NMR (300 MHz, CDCl_3): δ 7.43 (m, 3J = 8.5 Hz, 2 H), 7.24-7.36 (m, 10 H), 7.10 (d, 4J = 1.0 Hz, 1 H), 6.87-6.90 (m, 2 H), 6.10 (d, 4J = 1.0 Hz, 1 H), 5.55 (s, 2 H), 3.98-4.16 (m, 2 H), 1.68 (s, 3 H), 1.19 (t, 3J = 7.2 Hz, 3 H). ^{13}C NMR (300 MHz, CDCl_3): δ 166.3, 142.4, 139.9, 131.1, 137.6, 136.9, 131.4, 129.5, 128.8, 128.7, 128.6, 127.9, 127.8, 121.6, 111.4, 100.9, 69.4, 60.1, 59.8, 17.1, 14.3. HRMS (EI, $[\text{M}]^+$): calcd. for $\text{C}_{28}\text{H}_{26}\text{BrNO}_2$, 487.1147; found: 487.1164.

Ethyl 1-(diphenylmethyl)-5-methyl-2-phenyl-1,2-dihydropyridine-3-carboxylate (4d): Using the General Procedure from above (GP III).

Yellow crystals; purified by column chromatography over silica gel; 75 mg (37 % yield). $R_f = 0.44$ (hexane:diethylether 4:1). M. p. = 139 °C. IR (neat): 3027, 2925, 1688, 1526, 1171, 699 cm^{-1} . ^1H NMR (300 MHz, CDCl_3): δ 7.47 (m, $^3J = 7.9$ Hz, $^3J = 1.4$ Hz, 2 H), 7.25-7.38 (m, 8 H), 7.21-7.23 (m, 3 H), 7.12 (d, $^4J = 1.1$ Hz, 1 H), 6.84-6.86 (m, 2 H), 6.08 (d, $^4J = 0.9$ Hz, 1 H), 5.59 (s, 1 H), 5.57 (s, 1 H), 3.98-4.13 (m, 2 H), 1.67 (s, 3 H), 1.17 (t, $^3J = 7.2$ Hz, 3 H). ^{13}C NMR (300 MHz, CDCl_3): δ 166.4, 143.4, 140.2, 139.2, 137.6, 136.9, 129.6, 128.7, 128.5, 128.3, 127.81, 127.76, 127.69, 127.1, 11.7, 100.5, 69.0, 60.8, 59.7, 17.1, 14.3. HRMS (EI, $[\text{M}]^+$): calcd. for $\text{C}_{28}\text{H}_{27}\text{NO}_2$, 410.2114; found: 410.2100.

Ethyl 1-(diphenylmethyl)-5-methyl-2-(4-nitrophenyl)-1,2-dihydropyridine-3-carboxylate (4e): Using the General Procedure from above (GP III).

Red crystals; purified by column chromatography on silica gel; 97 mg (43 % yield). $R_f = 0.30$ (hexane : diethylether 4:1). M. p. = 129 °C. IR (neat): 3001, 2925, 1684, 1522, 1356, 1190, 851, 701 cm^{-1} . ^1H NMR (300 MHz, CDCl_3): δ 8.10-8.15 (m, $^3J = 8.8$ Hz, 2 H), 7.53-7.57 (m, $^3J = 8.8$ Hz, 2 H), 7.20-7.36 (m, 8 H), 7.12 (d, $^4J = 0.8$ Hz, 1 H), 6.92-6.95 (m, 2 H), 6.21 (d, $^4J = 1.0$ Hz, 1 H), 5.72 (d, $^4J = 0.7$ Hz, 1 H), 5.56 (s, 1 H), 4.01-4.18 (m, 2 H), 1.71 (d, $^4J = 0.7$ Hz, 3 H), 1.21 (t, $^3J = 7.2$ Hz, 3 H). ^{13}C NMR (300 MHz, CDCl_3): δ 166.1, 150.1, 147.3, 139.6, 138.8, 138.0, 137.2, 129.2, 129.1, 128.7, 128.6, 128.1, 128.0, 127.6, 123.6, 110.9, 102.0, 70.2, 60.1, 59.8, 17.1, 14.3. HRMS (EI, $[\text{M}]^+$): calcd. for $\text{C}_{28}\text{H}_{26}\text{N}_2\text{O}_4$, 454.1893; found: 454.1880.

Ethyl 2-(2,4-dichlorophenyl)-1-(diphenylmethyl)-5-methyl-1,2-dihydropyridine-3-carboxylate (4f): Using the General Procedure from above (GP III).

Yellow crystals; purified by column chromatography over silica gel; 127 mg (53 % yield). $R_f = 0.44$ (hexane: diethylether 4:1). M. p. = 171 °C. IR (Nujol): 2840, 1694, 1530, 1462, 1377, 1299, 1190, 667, 736, 698 cm^{-1} . ^1H NMR (300 MHz, CDCl_3): δ 7.79 (d, $^3J = 8.4$ Hz, 1 H), 7.25-7.42 (m, 7 H), 7.16-7.21 (m, 2 H), 6.67-6.70 (m, 4 H), 6.11 (s, 1 H), 5.97 ($^4J = 1.1$ Hz, 1 H), 5.58 (s, 1 H), 3.92-4.10 (m, 2 H), 1.67 (d, $^4J = 0.7$ Hz, 3 H), 1.14 (t, $^3J = 7.2$ Hz, 3 H). ^{13}C NMR (300 MHz, CDCl_3): δ 165.9, 140.2, 140.0, 139.1, 138.6, 136.5, 134.1, 131.9, 131.4, 129.3, 128.7, 128.5, 128.4, 128.3, 128.1, 127.8, 127.7, 112.4, 99.3, 68.6, 59.9, 57.1, 17.0, 14.2. HRMS (EI, $[\text{M}]^+$): calcd. for $\text{C}_{28}\text{H}_{25}\text{Cl}_2\text{NO}_2$, 477.1262; found: 477.1260. X-ray quality crystals were obtained by recrystallization from $\text{CH}_2\text{Cl}_2/\text{Et}_2\text{O}$.

Ethyl 1-(diphenylmethyl)-5-methyl-2-[2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl]-1,2-dihydropyridine-3-carboxylate (4g):

Using the General Procedure from above (GP III). Yellow oil; purified by column chromatography over silica gel; 51 mg (19 % yield). R_f = 0.33 (hexane:diethylether 4:1). IR (neat): 3025, 2934, 2723, 1690, 1531, 1312, 1189, 964, 862 cm^{-1} . ^1H NMR (300 MHz, CDCl_3): δ 8.03 (d, 3J = 7.9 Hz, 1 H), 7.62 (dd, 3J = 7.6 Hz, 4J = 1.2 Hz, 1 H), 7.48 (ddd, 3J = 7.6 Hz, 4J = 1.5 Hz, 1 H), 7.25-7.42 (m, 6 H), 7.10-7.17 (m, 4 H), 6.64-6.67 (m, 2 H), 6.21 (s, 1 H), 6.01 (4J = 0.8 Hz, 1 H), 5.87 (s, 1 H), 3.86-4.12 (m, 2 H), 1.97 (t, 3J = 7.2 Hz, 3 H), 1.66 (d, 4J = 0.8 Hz, 3 H), 1.20 (s, 6 H), 1.12 (s, 6 H). ^{13}C NMR (300 MHz, CDCl_3): δ 166.1, 150.3, 141.1, 140.5, 137.5, 137.1, 133.4, 131.6, 129.7, 129.3, 128.3, 128.2, 128.0, 127.3, 127.0, 126.97, 114.2, 98.3, 83.7, 67.3, 59.4, 25.1, 24.2, 17.1, 26.9. HRMS (ESI, $[\text{M}-\text{H}]^+$): calcd. for $\text{C}_{34}\text{H}_{37}\text{BNO}_4$, 534.2810; found: 534.2815.

Ethyl 1-(2-iodophenyl)-5-methyl-2-phenyl-1,2-dihydropyridine-3-carboxylate (4h):

Using the General Procedure from above (GP III). Yellow oil; purified by column chromatography on silica gel; 113 mg (51 % yield). R_f = 0.42 (hexane:diethylether 4:1). IR (neat): 2921, 1694, 1532, 1467, 1220, 1175, 1019, 758, 697 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 7.90 (dd, 3J = 7.9 Hz, 4J = 1.5 Hz, 1 H), 7.24-7.33 (m, 7 H), 7.17 (ddd, 3J = 7.5 Hz, 3J = 7.9 Hz, 4J = 1.5 Hz, 1 H), 6.91 (ddd, 3J = 7.3 Hz, 3J = 7.5 Hz, 4J = 1.5 Hz, 1 H), 6.85 (dd, 3J = 7.9 Hz, 4J = 1.3 Hz, 1 H), 6.34 (m, 4J = 1.1 Hz, 1 H), 5.85 (d, 4J = 1.1 Hz, 1 H), 4.16 (dq, 3J = 7.0 Hz, 4J = 1.8 Hz, 2 H), 1.88 (d, 4J = 1.1 Hz, 3 H), 1.26 (t, 3J = 7.0 Hz, 3 H). ^{13}C NMR (400 MHz, CDCl_3): δ 166.1, 147.9, 141.4, 140.4, 137.2, 136.7, 129.4, 129.1, 128.2, 128.0, 127.8, 127.1, 114.4, 104.9, 96.5, 61.2, 60.1, 16.9, 14.3. HRMS (EI, $[\text{M}]^+$): calcd. for $\text{C}_{21}\text{H}_{20}\text{NO}_2\text{I}$, 445.0538; found: 445.0530.

Ethyl 1-(2-bromophenyl)-5-methyl-2-phenyl-1,2-dihydropyridine-3-carboxylate (4i):

Using the General Procedure from above (GP III). Yellow oil; purified by column chromatography on silica gel; 73 mg (37 % yield). R_f = 0.36 (hexane:diethylether 4:1). IR (neat): 2852, 1696, 1463, 1377, 907, 735 cm^{-1} . ^1H NMR (300 MHz, CDCl_3): δ 7.59 (dd, 3J = 7.9 Hz, 4J = 1.6 Hz, 1 H), 7.22-7.33 (m, 7 H), 7.14 (ddd, 3J = 7.7 Hz, 4J = 1.6 Hz, 1 H), 7.03 (ddd, 3J = 7.7 Hz, 4J = 1.6 Hz, 1 H), 6.93 (dd, 3J = 7.9 Hz, 4J = 1.6 Hz, 1 H), 6.41 (m, 4J = 1.0 Hz, 1 H), 5.91 (d, 4J = 0.9 Hz, 1 H), 4.11-4.21 (m, 2 H), 1.85 (d, 4J = 1.0 Hz, 3 H), 1.25 (t, 3J = 7.0 Hz, 3 H). ^{13}C NMR (300 MHz, CDCl_3): δ 166.1, 144.7, 141.5, 137.1, 136.5, 134.0, 129.2, 128.25,

128.19, 127.7, 127.4, 126.9, 120.2, 114.4, 105.3, 60.7, 60.1, 17.0, 14.3. HRMS (EI, $[M]^+$): calcd. for $C_{21}H_{20}NO_2Br$, 397.0677; found: 397.0675.

Ethyl 1-(2-bromo-4-methoxyphenyl)-5-methyl-2-phenyl-1,2-dihydropyridine-3-carboxylate (4j): Using the General Procedure from above (**GP III**). Yellow oil; purified by column chromatography on silica gel; 88 mg (41 % yield). R_f = 0.33 (hexane: diethylether 4:1). IR (neat): 2925, 1694, 1599, 1537, 1493, 1377, 1039, 908, 735 cm^{-1} . 1H

NMR (300 MHz, $CDCl_3$): δ 7.21-7.33 (m, 6 H) 7.12 (d, 4J = 2.8 Hz, 1 H), 6.79 (d, 3J = 8.8 Hz, 1 H), 6.66 (dd, 3J = 8.8 Hz, 4J = 2.8 Hz, 1 H), 6.29 (m, 4J = 1.0 Hz, 1 H), 5.77 (d, 4J = 1.0 Hz, 1 H), 4.08-4.20 (m, 2 H), 3.76 (s, 3 H), 1.84 (d, 4J = 1.0 Hz, 3 H), 1.23 (t, 3J = 7.2 Hz, 3 H). ^{13}C NMR (300 MHz, $CDCl_3$): 166.2, 158.2, 141.9, 137.9, 137.8, 136.6, 130.4, 128.2, 127.7, 127.0, 121.6, 118.5, 114.1, 113.7, 104.0, 61.2, 60.0, 55.7, 16.9, 14.3. HRMS (EI, $[M]^+$): calcd. for $C_{22}H_{22}NO_3Br$, 427.0783; found: 427.0786.

Diethyl 1-(diphenylmethyl)-5-methyl-1,2-dihydropyridine-2,3-dicarboxylate (4k): Using the General Procedure from above (**GP III**). Yellow oil; purified by column chromatography over silica gel, 64 mg (63 % yield). R_f = 0.12 (hexane:diethylether 4:1). IR (neat): 3021, 1735, 1693, 1522,

1454, 1376, 1175, 1030, 745, 703 cm^{-1} . 1H NMR (300 MHz, $CDCl_3$): δ 1.22 (t, 3J = 7.2 Hz, 3 H), 1.23 (t, 3J = 7.2 Hz, 3 H), 1.69 (d, 4J = 1.0 Hz, 3 H), 4.08-4.22 (m, 4 H), 5.18 (d, 4J = 1.4 Hz, 1 H), 5.95 (s, 1 H), 6.07-6.08 (m, 4J = 1.0 Hz, 1 H), 7.11 (s, 1 H), 7.22-7.37 (m, 10 H). ^{13}C NMR (300 MHz, $CDCl_3$): δ 172.6, 166.1, 140.2, 139.2, 137.8, 136.8, 129.7, 128.7, 128.0, 127.8, 127.7, 106.7, 104.2, 70.9, 61.1, 59.9, 57.7, 17.2, 14.4, 14.1. HRMS (EI, $[M]^+$): calcd. for $C_{25}H_{27}NO_4$, 405.1940; found: 405.1944.

Ethyl 1-(diphenylmethyl)-2-(2-furyl)-5-methyl-1,2-dihydropyridine-3-carboxylate (4l): Using the General Procedure from above (**GP III**). Yellow oil; purified by column chromatography on silica gel; 77 mg (37 % yield). R_f = 0.16 (hexane:diethylether 9:1). IR (neat): 3028, 2925, 1689, 1524, 1455,

1377, 1294, 1263, 1095, 742, 703 cm^{-1} . 1H NMR (300 MHz, $CDCl_3$): δ 7.23-7.28 (m, 9 H), 7.19 (s, 1 H), 6.96-6.99 (m, 2 H), 6.29 (dd, 3J = 3.3 Hz, 3J = 3.1 Hz, 1 H), 6.15 (d, 3J = 3.3 Hz, 1 H), 5.93 (d, 4J = 0.8 Hz, 1 H), 5.90 (s, 1 H), 5.68 (s, 1 H), 4.09 (q, 3J = 7.2 Hz, 2 H), 1.69 (d, 4J = 0.8 Hz, 3 H), 1.19 (t, 3J = 7.2 Hz, 3 H). ^{13}C NMR (300 MHz, $CDCl_3$): δ 166.0, 155.4, 141.9, 140.5, 139.5, 138.5, 136.0, 129.5, 128.6, 128.5, 128.4, 127.8, 127.7, 110.2, 107.8, 107.4, 102.4, 69.9, 59.8, 53.8, 17.2, 14.3. HRMS (EI, $[M]^+$): calcd. for $C_{26}H_{25}NO_3$, 339.1834; found: 339.1836.

Ethyl 1-(diphenylmethyl)-2-(4-fluorophenyl)-5-phenyl-1,2-dihydropyridine-3-carboxylate (4m): Using the General Procedure from above (**GP III**). Yellow crystals; purified by column chromatography over silica gel; 110 mg (46 % yield). $R_f = 0.20$ (hexane:diethylether 9:1). M. p. = 137 °C. IR (Nujol): 2925, 1696, 636, 1600, 1533, 1455, 1218, 907, 736, 699 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 7.72 (d, $^4J = 1.3$ Hz, 1 H), 7.46-7.50 (m, $^3J = 8.6$ Hz, 2 H), 7.35-7.42 (m, 3 H), 7.22-7.33 (m, 7 H), 7.12-7.14 (m, $^3J = 7.3$ Hz, 2 H), 7.06-7.11 (m, $^3J = 7.3$ Hz, 1 H), 7.00-7.05 (m, $^3J = 8.6$ Hz, 2 H), 6.84-6.86 (m, 2 H), 6.78 (s, 1 H), 5.70 (s, 1 H), 5.63 (s, 1 H), 3.65 (s, 3 H). ^{13}C NMR (100 MHz, CDCl_3): δ 166.6, 162.7 (d, $^1J_{\text{C,F}} = 246$ Hz), 139.0, 138.84 (d, $^4J_{\text{C,F}} = 3.1$ Hz), 138.5, 138.2, 138.1, 133.8, 129.5, 129.0 (d, $^3J_{\text{C,F}} = 7.6$ Hz), 128.97, 128.8, 128.7, 128.3, 128.1, 127.8, 124.6, 122.8, 115.4 (d, $^2J_{\text{C,F}} = 21.5$ Hz), 112.9, 106.0, 69.3, 60.9, 51.3. HRMS (EI, $[\text{M}]^+$): calcd. for $\text{C}_{32}\text{H}_{26}\text{FNO}_2$, 475.1948; found: 475.1938.

5. Synthesis of the β -Aminoesters **3**

General Procedure for the synthesis of the β -Aminoesters **3** (**GP IV**):

The vinyloxirane **1a** (0.75 mmol, 117 mg, 1.5 equiv.) and the corresponding aldimine **2** (0.5 mmol, 1.0 equiv.) were dissolved in a solvent mixture of dry toluene (1.0 ml) and dry THF (1.0 ml). Afterwards the solution was cooled to 0 °C and $\text{Sc}(\text{OTf})_3$ was added under a N_2 -atmosphere. After stirring for 1.5 h at 0 °C, the reaction mixture was quenched with a sat. NaCl-solution (5.0 ml) at 0 °C. The aqueous phase was extracted with CH_2Cl_2 (3 x 15 ml) and the combined organic layers were dried over Na_2SO_4 , filtered and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel.

Ethyl (3E)-2-[[2-bromophenyl]amino](phenyl)methyl-4-methyl-5-oxopent-3-enoate (3i):

Using the General Procedure from above (GP IV). Pale yellow oil; purified by column chromatography over silica gel; 153 mg (74 % yield, d. r. = 1.9:1). R_f = 0.13 (hexane : diethylether 4:1). IR (neat): 3398, 3062, 2980, 2825, 1732, 1691, 1595, 1510, 1452, 1317, 1176, 1020, 744 cm^{-1} . Major diastereoisomer is marked with one asterisk,

overlaid signals are marked with two asterisks. ^1H NMR (300 MHz, CDCl_3): δ = 9.49 (s, 1 H), 9.42* (s, 1 H), 7.41* (dd, 3J = 7.9 Hz, 4J = 1.5 Hz, 1 H), 7.39 (dd, 3J = 7.9 Hz, 4J = 1.5 Hz, 1 H), 6.95-7.03** (m, 1 H), 7.25-7.33** (m, 5 H), 6.51- 6.57** (m, 1 H), 6.62-6.69** (m, 1 H), 6.43* (dd, 3J = 8.5 Hz, 4J = 1.5 Hz, 1 H), 6.39 (dd, 3J = 8.5 Hz, 4J = 1.5 Hz, 1 H), 5.62* (d, 3J = 7.8 Hz, 1 H), 5.17 (d, 3J = 6.4 Hz, 1 H), 4.9 (dd, 3J = 6.4 Hz, 1 H), 4.82* (dd, 3J = 7.8 Hz, 1 H), 4.17 (q, 3J = 7.2 Hz, 2 H), 4.09* (q, 3J = 7.2 Hz, 2 H), 3.97-4.02** (m, 1 H), 1.64 (d, 4J = 1.5 Hz, 3 H), 1.56* (d, 4J = 1.5 Hz, 3 H), 1.21* (t, 3J = 7.2 Hz, 3 H), 1.14 (t, 3J = 7.2 Hz, 3 H). ^{13}C NMR (100.5 MHz, CDCl_3): δ = 194.3*, 194.2, 170.6*, 169.9, 145.7*, 145.3, 143.2, 143.1*, 142.8, 142.3*, 139.1, 138.8*, 132.43*, 132.4, 128.8*, 128.4*, 128.3, 128.2*, 128.16, 126.8, 126.7*, 118.8, 118.6*, 113.0, 112.6*, 110.4, 110.3*, 61.9*, 61.8, 60.3, 59.8*, 52.9, 52.4*, 14.1*, 14.0, 9.6, 9.4*. HRMS (ESI, $[\text{M}-\text{H}]^+$): calcd. for $\text{C}_{34}\text{H}_{37}\text{BrNO}_4$; found: 534.2815.

Ethyl (3E)-4-methyl-5-oxo-2-(phenyl{[2-(trifluoromethyl)phenyl]amino}methyl)pent-3-enoate (3n):

Using the General Procedure from above (GP IV). Pale yellow oil, purified by column chromatography over silica gel; 171 mg (84 % yield, d. r. = 1.3:1). R_f = 0.12 (hexane: diethylether 4:1). IR (neat): 3420, 3060, 2983, 1731, 1693, 1614, 1588, 1329, 1288, 1102, 1033, 749 cm^{-1} . Major diastereoisomer is marked with one asterisk, overlaid signals are marked with two asterisks. ^{19}F NMR (282

MHz): δ = -62.9*, -62.8. ^1H NMR (300 MHz, CDCl_3): δ = 9.47* (s, CHO, 1 H), 9.41 (s, CHO, 1 H), 7.41-7.46** (m, 1 H), 7.27-7.34** (m, 5 H), 7.14-7.24** (m, 1 H), 6.60-6.72** (m, 2 H), 6.49 (d, 3J = 8.3 Hz, 1 H), 6.47* (d, 3J = 8.4 Hz, 1 H), 5.76 (d, 3J = 7.3 Hz, 1 H), 5.21* (d, 3J = 6.0 Hz, 1 H), 4.92* (dd, 3J = 6.0 Hz, 1 H), 4.82 (dd, 3J = 7.3 Hz, 1 H), 4.11-4.18** (m, 2 H), 3.94-4.09** (m, 1 H), 1.63* (d, 4J = 1.5 Hz, 3 H), 1.57 (d, 4J = 1.5 Hz, 3 H), 1.19 (t, 3J = 7.0 Hz, 3 H), 1.15* (t, 3J = 7.0 Hz, 3 H). ^{13}C NMR (75.4 MHz, CDCl_3): δ = 194.2, 194.1*, 170.5, 169.7*, 145.5, 144.9*, 143.7**, 142.8*, 142.2, 138.9*, 138.8, 133.0, 132.9*, 128.9**, 128.3*, 128.2, 126.7*, 126.6** (q, $^3J_{\text{CF}}$ = 5.5 Hz, 1 C), 126.5, 125.4** (q, $^1J_{\text{CF}}$ = 272.3 Hz, 1 C), 117.0*, 116.8, 114.3* (q, $^1J_{\text{CF}}$ = 29.3 Hz, 1 C), 114.1 (q, $^1J_{\text{CF}}$ = 29.3 Hz, 1 C), 113.5*, 112.9, 61.9, 61.8*, 59.6*, 59.2, 52.7*, 52.4, 13.9**, 9.4*, 9.3. HRMS (ESI, $[\text{M}+\text{Na}]^+$): calcd. for $\text{C}_{22}\text{H}_{22}\text{F}_3\text{NO}_3\text{Na}$, 428.1443; found: 428.1447.

B. Brunner, CDC13, BB023

Data Collected on:
mercury300-mercury300
Archive directory:
/home/bbrunner/vnmrSYS/data
Sample directory:

File: bb023clear

Pulse Sequence: s2pul
Solvent: CDC13

Temp. 25.0 C / 298.1 K
Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.411 sec
Width 4803.1 Hz
16 repetitions

OBSERVE H1, 300.0510801 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min

¹³C OBSERVE

Data Collected on:
mercury300-mercury300
Archive directory:
/home/bbrunner/vnmrsys/data
Sample directory:

File: bb023carbon

Pulse Sequence: s2pul
Solvent: CDCl₃

Temp. 25.0 C / 298.1 K
Operator: bbrunner

Pulse 30.0 degrees
Acq. time 1.000 sec
Width 18867.9 Hz
30000 repetitions

OBSERVE C13, 75.4479044 MHz
DECOUPLE H1, 300.0525807 MHz
Power 37 dB

continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Ft size 131072
Total time 9 hr, 52 min

B. Brunner, CDC13, BB080

Data Collected on:
nmr2-mercury400
Archive directory:
/export/home/bbrunner/vnmrsys/data
Sample directory:

File: PROTON

Pulse Sequence: s2pul
Solvent: CDC13

Temp. 25.0 C / 298.1 K
Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.664 sec
Width 7199.4 Hz
16 repetitions

OBSERVE F1, 399.7532481 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Ft size 65536
Total time 1 min

B. Brunner, CDCl₃, BB080carbon

Data Collected on:

nmr2-mercury400

Archive directory:

/export/home/bbrunner/vnmrsws/data

Sample directory:

File: CARBON

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

Operator: bbrunner

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25125.6 Hz

15000 repetitions

OBSERVE C13, 100.5180380 MHz

DECOUPLE H1, 399.7552131 MHz

Power 42 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 11 hr, 15 min

Data Collected on:
pochacco-inova500
Archive directory:
/home/bbrunner/vnmrsys/data
Sample directory:

File: 20060324-bb067_Proton-001

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Sample #1

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.403 sec
Width 4803.1 Hz
16 repetitions

OBSERVE H1, 300.0510850 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Ft size 65536
Total time 1 min

Data Collected on:
pochacco-inova500
Archive directory:

Sample directory:

File: 20060324-bb067_s2pul-001

Pulse Sequence: s2pul

Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Sample #1, user 1-14-87

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.809 sec

Width 18115.9 Hz

2000 repetitions

OBSERVE C13, 75.4479051 MHz

DECOUPLE H1, 300.0525807 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 1 hr, 43 min

Data Collected on:
mercury300-mercury300
Archive directory:
/export/home/bbrunner/vnmrsys/data
Sample directory:

File: 20060322-bb051_Proton-001

Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 25.0 C / 298.1 K
Sample #31, Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.403 sec
Width 4803.1 Hz
16 repetitions

OBSERVE H1, 300.0510833 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min

Data Collected on:
mercury300-mercury300
Archive directory:

Sample directory:

File: 20060322-bb051_s2pul-001

Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 25.0 C / 298.1 K
Sample #31, Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.809 sec
Width 18115.9 Hz
1000 repetitions

OBSERVE C13, 75.4479047 MHz
DECOUPLE H1, 300.0525807 MHz
Power 36 dB

continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Ft size 131072
Total time 51 min

Data Collected on:
mercury300-mercury300
Archive directory:
/export/home/bbrunner/vnmrSYS/data
Sample directory:

File: 20060121-bb146_Proton-001

Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 25.0 C / 298.1 K
Sample #3, Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.403 sec
Width 4803.1 Hz
16 repetitions

OBSERVE H1, 300.0510858 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min

Data Collected on:
mercury300-mercury300
Archive directory:

Sample directory:

File: 20060121-bb146_Carbon-001

Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 25.0 C / 298.1 K
Sample #10, Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.809 sec
Width 18115.9 Hz

2000 repetitions
OBSERVE C13, 75.4479053 MHz
DECOUPLE H1, 300.0525807 MHz

Power 36 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 43 min

2f

```
Data Collected on:
mercury300-mercury300
Archive directory:
/export/home/bbrunner/vmrsys/data
Sample directory:
```

File: PROTON

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.409 sec
Width 4800.8 Hz
16 repetitions
OBSERVE H1, 300.0510748 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min

B. Brunner, CDC13, BB054

Data Collected on:
mercury300-mercury300
Archive directory:
/export/home/bbrunner/vnmrsys/data
Sample directory:

File: CARBON

Pulse Sequence: s2pul
Solvent: CDC13

Temp. 25.0 C / 298.1 K
Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.000 sec
Width 18867.9 Hz
10000 repetitions

OBSERVE C13, 75.4479057 MHz
DECOUPLE H1, 300.0525807 MHz
Power 37 dB

continuously on
WALTZ-16 modulated
DATA PROCESSING

Line broadening 1.0 Hz
Ft size 131072
Total time 9 hr, 13 min

```
Data Collected on:
mercury300-mercury300
Archive directory:
/export/home/bbrunner/
Sample directory:
```

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Sample #38, Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.404 sec
Width 4800.8 Hz
16 repetitions
OBSERVE H1, 300.0510840 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Ft size 65536
Total time 1 min

Data Collected on:
mercury300-mercury300
Archive directory:

Sample directory:

File: bbrunner-20051206-_Carbon-001

Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Sample #27, Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.809 sec
Width 18115.9 Hz

5000 repetitions

OBSERVE C13, 75.4479050 MHz
DECOUPLE H1, 300.0525807 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 4 hr, 18 min

Data Collected on:
 mercury300-mercury300
 Archive directory:
 /export/home/bbrunner/vnmrsys/data
 Sample directory:

File: 20060410-bb061_Proton-001

Pulse Sequence: s2pul
 Solvent: cdcl3

Temp. 25.0 C / 298.1 K
 Sample #2, Operator: bbrunner

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 3.403 sec
 Width 4803.1 Hz
 16 repetitions

OBSERVE H1, 300.0510864 MHz

DATA PROCESSING
 Line broadening 0.2 Hz
 F1 size 65536
 Total time 1 min

Data Collected on:
mercury300-mercury300
Archive directory:

Sample directory:

File: 20060410-bb061_s2pul-001

Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Sample #2, Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.809 sec
Width 18115.9 Hz
2000 repetitions

OBSERVE C13, 75.4479069 MHz
DECOUPLE H1, 300.0525807 MHz
Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 1 hr, 43 min

Data Collected on:
mercury300-mercury300
Archive directory:
/export/home/bbrunner/vnmrsvs/data
Sample directory:

File: 20060322-bb043_Proton-001

Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 25.0 C / 298.1 K
Sample #30, Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.403 sec
Width 4803.1 Hz
16 repetitions

OBSERVE H1, 300.0510837 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Ft size 65536
Total time 1 min

Data Collected on:
mercury300-mercury300
Archive directory:

Sample directory:

File: 20060328-bb043_s2pul-001

Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Sample #1, Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.809 sec
Width 18115.9 Hz
5000 repetitions

OBSERVE C13, 75.4479029 MHz
DECOUPLE H1, 300.0525807 MHz
Power 36 dB

continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Ft size 131072
Total time 4 hr, 18 min

B. Brunner, CDCl₃, BB071

Data Collected on:
nmr2-mercury400
Archive directory:
/export/home/bbrunner/vnmr/ays/data
Sample directory:

File: bb071

Pulse Sequence: s2pul
Solvent: CDCl₃

Temp. 25.0 C / 298.1 K
Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.664 sec
Width 7199.4 Hz
16 repetitions

OBSERVE H1, 399.7727846 MHz

DATA PROCESSING

Line broadening 0.2 Hz
FT size 65536

Total time 1 min

4a

13C OBSERVE

Data Collected on:
mercury300-mercury300
Archive directory:
/export/home/bbrunner/vnmrsys/data
Sample directory:

File: bb071carbon

Pulse Sequence: s2pul
Solvent: CDCl3

Temp. 25.0 C / 298.1 K
Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.000 sec
Width 18867.9 Hz
12000 repetitions

OBSERVE C13, 75.4479028 MHz
DECOUPLE H1, 300.0525807 MHz

Power 37 dB

continuously on
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 11 hr, 4 min

B. Brunner, CDC13, BB047

Data Collected on:
mercury300-mercury300
Archive directory:
/home/bbrunner/vnmrSYS/data
Sample directory:

File: bb047

Pulse Sequence: s2pul
Solvent: CDC13

Temp. 25.0 C / 298.1 K
Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.411 sec
Width 4803.1 Hz
16 repetitions

OBSERVE H1, 300.0510982 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min

13C OBSERVE

Data Collected on:
mercury300-mercury300
Archive directory:
/home/bbrunner/vnmrsys/data
Sample directory:

File: bb047carbon

Pulse Sequence: s2pul
Solvent: CDCl3

Temp. 25.0 C / 298.1 K
Operator: bbrunner

Pulse 30.0 degrees
Acq. time 1.000 sec
Width 18867.9 Hz
30000 repetitions
OBSERVE C13, 75.4479028 MHz
DECOUPLE H1, 300.0525807 MHz
Power 37 dB

continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Ft size 131072
Total time 9 hr, 52 min

B. Brunner, CDCl3, BB056

Data Collected on:
mercury300-mercury300
Archive directory:
/export/home/bbrunner/vnmrsys/data
Sample directory:

File: PROTON

Pulse Sequence: s2pul
Solvent: CDCl3

Temp. 25.0 C / 298.1 K
Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.409 sec
Width 4800.8 Hz
16 repetitions

OBSERVE H1, 300.0510742 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min

B. Brunner, CDCl₃, BB056

Data Collected on:
mercury300-mercury300
Archive directory:
/export/home/bbrunner/vnmr/ays/data
Sample directory:

File: CARBON

Pulse Sequence: s2pul
Solvent: CDCl₃

Temp. 25.0 C / 298.1 K
Operator: bbrunner

Pulse 30.0 degrees
Acq. time 1.000 sec
Width 18867.9 Hz
30000 repetitions
OBSERVE C13, 75.4479028 MHz
DECOUPLE H1, 300.0525807 MHz
Power 37 dB
continuously on

WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 9 hr, 52 min

B. Brunner, CDC13, BB104

Data Collected on:
nmr2-mercury400
Archive directory:
/export/home/bbrunner/vmarsys/data
Sample directory:

File: PROTON

Pulse Sequence: s2pul
Solvent: CDC13

Temp. 25.0 C / 298.1 K
Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.664 sec
Width 7199.4 Hz
16 repetitions

OBSERVE H1, 399.7532539 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Ft size 65536
Total time 1 min

4d

B. Brunner, CDCl₃, BB104carbon

Data Collected on:

mercury300-mercury300

Archive directory:

/export/home/bbrunner/vmrsys/data

Sample directory:

File: CARBON

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

Operator: bbrunner

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.000 sec

Width 18867.9 Hz

10000 repetitions

OBSERVE C13, 75.4479050 MHz

DECOUPLE H1, 300.0525807 MHz

Power 37 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 9 hr, 13 min

4d

Data Collected on:
 mercury300-mercury300
 Archive directory:
 /export/home/bbrunner/vnmrsys/data
 Sample directory:

File: 20060213-bb158a_Proton-001

Pulse Sequence: s2pul
 Solvent: cdcl3

Temp. 25.0 C / 298.1 K
 Sample #1, Operator: bbrunner

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 3.403 sec
 Width 4803.1 Hz
 16 repetitions

OBSERVE H1, 300.0510823 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 1 min

Data Collected on:
mercury300-mercury300
Archive directory:

Sample directory:

File: 20060215-bb158_Carbon-001

Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 25.0 C / 298.1 K
Sample #1, Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.809 sec
Width 18115.9 Hz
5000 repetitions
OBSERVE C13, 75.4479058 MHz
DECOUPLE H1, 300.0525807 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Ft size 131072
Total time 4 hr, 18 min

Data Collected on:
 mercury300-mercury300
 Archive directory:
 /export/home/bbrunner/vnmrsys/data
 Sample directory:

File: 20060125-bb149_Proton-001

Pulse Sequence: s2pul
 Solvent: cdcl3

Temp. 25.0 C / 298.1 K
 Sample #24, Operator: bbrunner

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 3.403 sec
 Width 4803.1 Hz
 16 repetitions

OBSERVE H1, 300.0510820 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 1 min

Data Collected on:
mercury300-mercury300
Archive directory:

Sample directory:

File: 20060125-bb149_Carbon-001

Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 25.0 C / 298.1 K
Sample #24, Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.809 sec
Width 18115.9 Hz
5000 repetitions
OBSERVE C13, 75.4479045 MHz
DECOUPLE H1, 300.0525807 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Ft size 131072
Total time 4 hr, 18 min

4f

Data Collected on:
 mercury300-mercury300
 Archive directory:
 /export/home/bbrunner/vnmrsws/data
 Sample directory:

File: 20060117-bb144_Proton-001

Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
 Sample #46, Operator: bbrunner

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 3.403 sec
 Width 4803.1 Hz
 16 repetitions
 OBSERVE H1, 300.0510808 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 1 min

Data Collected on:
mercury300-mercury300
Archive directory:

Sample directory:

File: 20060122-bb144_Carbon-001

Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 25.0 C / 298.1 K
Sample #1, Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.809 sec
Width 18115.9 Hz
5000 repetitions
OBSERVE C13, 75.4479050 MHz
DECOUPLE H1, 300.0525807 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 4 hr, 18 min

B. Brunner, CDCl3, BB025

Data Collected on:

nmr2-mercury400

Archive directory:

/export/home/bbrunner/vnmrsys/data

Sample directory:

File: bb025

Pulse Sequence: s2pul

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

Operator: bbrunner

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.664 sec

Width 7199.4 Hz

16 repetitions

OBSERVE H1, 399.7858198 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min

4h

B. Brunner, CDCl3, BB025cb

Data Collected on:
nmr2-mercury400
Archive directory:
/export/home/bbrunner/vnmrsys/data
Sample directory:

File: bb025cb

Pulse Sequence: s2pul

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

Operator: bbrunner

Pulse 30.0 degrees
Acq. time 1.000 sec
Width 25125.6 Hz
30000 repetitions
OBSERVE C13, 100.5262239 MHz
DECOUPLE H1, 399.7877671 MHz

Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Ft size 65536
Total time 11 hr, 56 min

Data Collected on:
mercury300-mercury300
Archive directory:
/export/home/bbrunner/vnmrsys/data
Sample directory:

File: bbrunner-20051111-bb115_Proton-001

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Sample #25, Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.404 sec
Width 4800.8 Hz
16 repetitions
OBSERVE H1, 300.0510822 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Ft size 65536
Total time 1 min

B. Brunner, CDC13, BB115carbon

Data Collected on:
nmr2-mercury400
Archive directory:
/export/home/bbrunner/vnmrsys/data
Sample directory:

File: CARBON

Pulse Sequence: s2pul
Solvent: CDC13

Temp. 25.0 C / 298.1 K
Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
3000 repetitions

OBSERVE C13, 100.5180350 MHz
DECOUPLE H1, 399.7552131 MHz
Power 42 dB

continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 1.0 Hz
Ft size 65536
Total time 2 hr, 15 min

4i

Data Collected on:
mercury300-mercury300

Archive directory:
/export/home/bbrunner/vnmrSYS/data
Sample directory:

File: bbrunner-20051202-bb131_Proton-001

Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Sample #9, Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.403 sec
Width 4803.1 Hz
16 repetitions

OBSERVE H1, 300.0510802 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min

B. Brunner, CDCl3, BB131

Data Collected on:
nmr2-mercury400
Archive directory:
/export/home/bbrunner/vnmrSYS/data
Sample directory:

File: CARBON

Pulse Sequence: s2pul
Solvent: CDCl3

Temp. 25.0 C / 298.1 K
Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
1024 repetitions
OBSERVE C13, 100.5147580 MHz
DECOUPLE H1, 399.7421666 MHz
Power 42 dB
continuously on

WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 46 min

4j

Data Collected on:
 mercury300-mercury300
 Archive directory:
 /export/home/bbrunner/vnmrsys/data
 Sample directory:

File: 20060202-bb153_Proton-001

Pulse Sequence: s2pul
 Solvent: cdcl3

Temp. 25.0 C / 298.1 K
 Sample #20, Operator: bbrunner

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 3.403 sec
 Width 4803.1 Hz
 16 repetitions

OBSERVE H1, 300.0510812 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 1 min

Data Collected on:
mercury300-mercury300
Archive directory:

Sample directory:

File: 20060203-bb153_Carbon-001

Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Sample #37, Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.809 sec
Width 18115.9 Hz

5000 repetitions
OBSERVE C13, 75.4479045 MHz
DECOUPLE H1, 300.0525807 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 4 hr, 18 min

Data Collected on:
mercury300-mercury300
Archive directory:

Sample directory:

File: bbrunner-20051114-bb103carbon_Carbon-001

Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 25.0 C / 298.1 K
Sample #22, Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.809 sec
Width 18115.9 Hz

256 repetitions
OBSERVE C13, 75.4479058 MHz
DECOUPLE H1, 300.0525807 MHz
Power 36 dB

continuously on
WALTZ-16 modulated
DATA PROCESSING

Line broadening 1.0 Hz
FT size 131072
Total time 13 min

Data Collected on:
 mercury300-mercury300
 Archive directory:
 /export/home/bbrunner/vnmrSYS/data
 Sample directory:

File: bbrunner-20051114-bb103_Proton-001

Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
 Sample #20, Operator: bbrunner

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 3.404 sec
 Width 4800.8 Hz
 16 repetitions
 OBSERVE H1, 300.0510837 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 1 min

4l

B. Brunner, CDCl3, BB082

Data Collected on:
nmr2-mercury400
Archive directory:
/export/home/bbrunner/vnmrsys/data
Sample directory:

File: bb082

Pulse Sequence: s2pul
Solvent: CDCl3

Temp. 25.0 C / 298.1 K
Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.664 sec
Width 7199.4 Hz
16 repetitions

OBSERVE H1, 399.7532481 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min

¹³C OBSERVE

Data Collected on:
nmr2-mercury400
Archive directory:
/export/home/bbrunner/vnmrsys/data
Sample directory:

File: bb082carbon

Pulse Sequence: s2pul
Solvent: CDCl₃

Temp. 25.0 C / 298.1 K
Operator: bbrunner

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
15000 repetitions

OBSERVE C13, 100.5180342 MHz
DECOUPLE H1, 399.7552131 MHz

Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 11 hr, 15 min

Data Collected on:
 mercury300-mercury300
 Archive directory:
 /export/home/bbrunner/vnmrsys/data
 Sample directory:

File: 20060331-bb133f1_Proton-001

Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
 Sample #6, Operator: bbrunner

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 3.403 sec
 Width 4803.1 Hz
 16 repetitions
 OBSERVE H1, 300.0510821 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 1 min

Data Collected on:
mercury300-mercury300
Archive directory:

Sample directory:

File: 20060401-bb133f1_s2pul-001

Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 25.0 C / 298.1 K

Sample #2, Operator: bbrunner

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.809 sec

Width 18115.9 Hz

5000 repetitions

OBSERVE C13, 75.4479053 MHz

DECOUPLE H1, 300.0525807 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 4 hr, 18 min

Bernard Brunner

Data Collected on:
mercury300-mercury300
Archive directory:

Sample directory:

File: 20051203-bb730a_Proton_-001

Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 25.0 C / 298.1 K
Operator: vnmr1

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.268 sec
Width 6410.3 Hz
8 repetitions

OBSERVE H1, 399.6867960 MHz

DATA PROCESSING

Line broadening 0.1 Hz

Ft size 65536

Total time 0 min

bbrunner
bb730a ki4332
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Archive directory:
Sample directory:
File: CARBON

Pulse Sequence: s2pul

Solvent: cdcl3

Temp. 25.0 C / 298 K

Operator: vnmr15

VNMR5-400 "vnmr400"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.337 sec

Width 24509.8 Hz

4096 repetitions

OBSERVE C13, 100.5013233 MHz

DECOUPLE H1, 399.688004 MHz

Power 41 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

Total time 2 hr, 40 min, 13 sec

Bernard Brunner

Archive directory:
Sample directory:
File: ROESY

Pulse Sequence: ROESY
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: vnmr1
VNMR-400 "vnmr400"

Relax. delay 1.500 sec
Mixing 0.300 sec
Acq. time 0.246 sec
Width 4166.7 Hz
2D Width 4166.7 Hz
32 repetitions
2 x 256 increments

OBSERVE F1, 399.6868017 MHz
DATA PROCESSING
Gauss apodization 0.114 sec
F1 DATA PROCESSING
Gauss apodization 0.113 sec
Ft size 4096 x 4096
Total time 9 hr, 32 min, 12 sec