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Supporting Information

69451 Weinheim, Germany

Branched-Regioselective Hydroformylation with Catalytic Amounts of a Reversibly Bound Directing Group

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Supporting Information

I General

All reactions were carried out in oven-dried glassware under an atmosphere of argon (Argon 5.0 from Sauerstoffwerke Friedrichshafen). All reagents were obtained commercially unless otherwise noted. 3-Buten-1-ol was purchased from Aldrich, cis-3-Hexen-1-ol and trans-3-Hexen-1-ol from ABCR. All solvents were dried and distilled by standard procedures. Chromatographic purification of products was accomplished using flash chromatography[1] on Machery-Nagel silica gel 60® (230-400 mesh). Hydroformylation reactions were performed in an Endeavor parallel autoclave with 8 reaction vessels from Argonaut Technologies. Gases: Carbon monoxide 3.7, hydrogen 4.3 (1:1, Messer-Griesheim).

Achiral GC analyses were performed on an Agilent 6890N apparatus equipped with a Supelcowax 10 column (30 m x 0.25 mm ID x 0.25 μm film).

Nuclear magnetic resonance spectra were acquired on a Bruker Avance 400 spectrometer (400 MHz and 100 MHz for ^{1}H and ^{13}C respectively), on a Bruker Avance DRX 250 spectrometer and on a Varian Mercury 300 spectrometer. NMR-spectra are referenced internally according to TMS [0.00 ppm (^{1}H)] or residual protio solvent signals [77.10 ppm for $CDCl_3$ (^{13}C)].[2] Data for ^{1}H-NMR are reported as follows: chemical shift (δ in ppm), multiplicity (s, singlet; d, doublet; t, triplet; q, quartet; quint, quintet; sext, sextet; hept, heptet; oct, octet; m, multiplet; m_c, symmetrical multiplet; br, broad

signal; *p*, pseudo), integration, coupling constant (Hz). Data for ^{13}C-NMR are reported in terms of chemical shift, integration, coupling constant (Hz). High-resolution mass spectra were obtained on a Finnigan MAT 8200 instrument (EI: 70 eV; CI/NH_3: 110 eV).

II Experimental procedures and characterizations

General procedure (g. p. 1) for the synthesis of (*E*)-alk-3-enoic acids or (*E*)-alk-3-enoic acid-methylesters from aldehydes by a deconjugative Knoevenagel reaction.[3]

Piperidine (40 µl, 34 mg, 0.40 mmol, 0.02 eq.) and acetic acid (23 µl, 0.40 mmol, 0.02 eq.) was stirred for 5 min. in DMSO (1ml). Afterwards DMSO (20 ml), Malonic acid (4.16 g, 40.0 mmol, 2.0 eq.) [or Malonic acid monomethylester (4.18 ml, 4.72 g, 40.0 mmol) to yield the (*E*)-alk-3-enoic acid-methylesters] and the aldehyde (20 mmol) were added and the reaction mixture was stirred under argon at RT for 20 min. Then the solution was heated to 100 °C for 4-12 h. The solution was cooled to RT and water (40 ml) and diethyl ether (40 ml) were added. The phases were separated and the aqueous phase was extracted with diethyl ether (3 x 40 ml). The combined organic phases were washed with water (3 x 30 ml), dried over Na_2SO_4, filtrated and the solvent was evaporated under reduced pressure. The crude product was purified by column chromatography (ethyl acetate/ cyclohexane).

General procedure (g. p. 2) for the synthesis of homoallylic alcohols from (*E*)-alk-3-enoic acids.

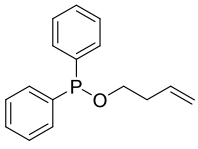
The β,γ-unsaturated acid (9.0 mmol, 1.0 eq.) was dissolved in THF (5 ml), and this solution was added to a suspension of $LiAlH_4$ (9.0 mmol, 1.0 eq.) in THF (5 ml) at 0 °C. The reaction mixture was allowed to warm to RT and was stirred for further 2 h. The reaction was quenched by addition of aqueous saturated Na_2SO_4 (until the reaction mixture turned white) and additional Na_2SO_4 (solid) was added. The precipitate was filtered off and washed with diethyl ether. The solvent of the filtrate was removed under reduced pressure. The crude product was purified by column chromatography (ethyl acetate/ cyclohexane).

General procedure (g. p. 3) for the synthesis of γ-lactones from homoallylic alcohols (Hydroformylation and PCC oxidation).

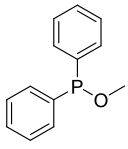
The oven dried glass inlet of the autoclave was equipped with molecular sieves (4 Å) and washed with argon. Homoallylic alcohol (0.6 mmol) was added under argon to the glass inlet and dried over the molecular sieves for 30 min. During this time a solution of the ligand $Ph_2P(OMe)$ (13.0 mg, 0.06 mmol) in THF (3 ml) was prepared. To this solution $Rh(CO)_2acac$ (1.5 mg, 0.006 mmol) was added and the mixture was stirred for 5 min. This mixture was transferred into the glass inlet equipped with the homoallylic alcohol and the molecular sieves. The glass inlet was transferred into the autoclave. The autoclave was sealed and purged three times with 5 bar of the CO/H_2 gas-mixture (1:1). The autoclave was pressurized with 5 bar of CO/H_2 (1:1), heated to 40 °C and pressurized with 20 bar of CO/H_2 (1:1). The reaction mixture was stirred at 40 °C for 12 h.

After pressure release, a sample was taken for GC-analysis. The solvent of the crude reaction mixture was evaporated. The residue was dissolved in CH_2Cl_2 (5 ml) and Al_2O_3 (1.2 g), PCC (256 mg, 1.2 mmol, 2.0 eq.) and NaOAc (25 mg, 0.3 mmol) were added. The reaction mixture was stirred at RT for 12 h, and subsequently filtered through a plug of silica gel. The silica gel was washed with CH_2Cl_2 (70 ml) and for more polar substrates with additional EtOAc (70 ml). The solvent of the filtrate was evaporated and the γ-lactone could be obtained analytically pure without further purification.

Synthesis of Homoallyl-diphenylphosphinite (2) and Methyl-diphenylphosphinite (3)



2



3

The alcohol (5.0 mmol) was dissolved in Et_2O (30 ml). The reaction mixture was cooled to 0 °C and NEt_3 (0.69 ml, 0.51 g, 5.0 mmol) was added. Afterwards $ClPPh_2$ (0.90 ml, 1.1 g, 5.0 mmol) was added over a period of 10 min. The reaction mixture was allowed to warm up to RT and was filtrated under argon over a plug of Al_2O_3 (neutral). The precipitate was washed with Et_2O (80 ml) and the solvent of combined filtrates was removed in vacuo to give the analytically pure title compounds (1.16 g, 91% for **2**, 919mg, 85% for **3**).

Analytical data for **2:**

^{1}H-NMR (400.130 MHz, $CDCl_3$): δ = 2.43 (m_c, 2H, 2-H), 3.89 (dt, *J* = 9.4, 6.8 Hz, 2H, 1-H), 5.05 (m_c, 2H, 4-H), 5.81 (ddt, *J* = 17.1, 10.3, 6.8 Hz, 3-H), 7.26-7.36 (m, 6H, Ar-H), 7.45-7.52 (m, 4H, Ar-H).

^{13}C-NMR (100.613 MHz, $CDCl_3$): δ = 36.0 (d, *J* = 7.5 Hz, 1C), 69.5 (d, *J* = 19.6 Hz, 1C), 117.0 (s, 1C), 128.3 (d, *J* = 7.0 Hz, 4C), 129.3 (s, 2C), 130.4 (d, *J* = 21.5 Hz, 4C), 134.6 (s, 1C), 142.1 (d, *J* = 17.9 Hz, 2C).

^{31}P-NMR (161.976 MHz, $CDCl_3$): δ = 112.4.

HRMS (CI, NH_3): calculated $(M+H)^+$: 257.109528, found: 257.109701.

Analytical data for **3:**

^{1}H-NMR (250.130 MHz, C_6D_6): δ = 3.39 (d, *J* = 13.8 Hz, 3H, OMe), 6.98-7.18 (m, 6H, Ar-H), 7.52-7.62 (m, 4H, Ar-H).

^{13}C-NMR (62.895 MHz, C_6D_6): δ = 56.4 (d, *J* = 19.9 Hz, 1C, OMe), 128.6 (d, *J* = 6.4 Hz, 4C), 129.4 (2C), 130.7 (d, *J* = 22.0 Hz, 4C), 142.6 (d, *J* = 19.3 Hz, 2C).

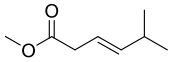
^{31}P-NMR (101.253 MHz, C_6D_6): δ = 117.0.

Analytical data for **3** were identical to those reported previously.[4]

Substrate synthesis

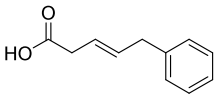
Synthesis of (*E*)-Alk-3-enoic acids

Synthesis of (*E*)-5-Methyl-hex-3-enoic acid methyl ester



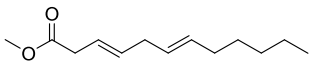
According to general procedure 1, (*E*)-5-Methyl-hex-3-enoic acid methyl ester (1.65 g, 58%) was obtained from commercially available Isovaleraldehyde and Malonic acid monomethylester. The analytical data were identical to those reported previously.[5]

Synthesis of (*E*)-5-Phenyl-pent-3-enoic acid



According to general procedure 1, (*E*)-5-Phenyl-pent-3-enoic acid (2.60 g, 74%) was obtained from commercially available 3-Phenylpropionaldehyde and Malonic acid. The analytical data were identical to those reported previously.[6]

Synthesis of (*E,E*)-Dodeca-3,6-dienoic acid methyl ester

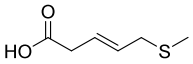


According to general procedure 1, (*E,E*)-Dodeca-3,6-dienoic acid methyl ester (3.36 g, 80%) was obtained from commercially available *trans*-4-Decenal and Malonic acid monomethylester.

^{1}H-NMR (400.130 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 7.1 Hz, 3H, CH_3), 1.18-1.42 (m, 6H), 1.9-2.5 (m, 2H), 2.72 (m_c, 2H), 3.02-3.07 (m, 2H), 3.68 (s, 3H, OCH_3), 5.32-5.62 (m, 4H).

^{13}C-NMR (100.613 MHz, $CDCl_3$): δ = 14.1, 22.6, 29.2, 31.5, 32.6, 35.6, 38.0, 51.8, 122.1, 127.6, 131.9, 133.4, 172.6.

Synthesis of (*E*)-5-Methylsulfanyl-pent-3-enoic acid

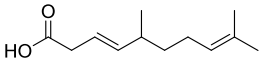


According to general procedure 1, (*E*)-5-Methylsulfanyl-pent-3-enoic acid (1.81 g, 62%) was obtained from commercially available 3-(Methylthio)-propanal and Malonic acid.

^{1}H-NMR (400.130 MHz, $CDCl_3$): δ = 2.02 (s, 3H, SCH_3), 3.08-3.12 (m, 2H), 3.13-3.17 (m, 2H), 5.52-5.70 (m, 2H).

^{13}C-NMR (100.613 MHz, $CDCl_3$): δ = 14.4, 35.7, 37.3, 123.9, 130.7, 177.7.

Synthesis of (*E*)-5,9-Dimethyl-deca-3,8-dienoic acid

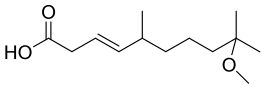


According to general procedure 1, (*E*)-5,9-Dimethyl-deca-3,8-dienoic acid (3.18 g, 81%) was obtained from commercially available Citronellal and Malonic acid.

^{1}H-NMR (400.130 MHz, $CDCl_3$): δ = 0.98 (d, *J* = 6.7 Hz, 3H), 1.18-1.34 (m, 2H), 1.58 (d, *J* = 0.9 Hz, 3H), 1.68 (d, *J* = 1.3 Hz, 3H), 1.90-2.03 (m, 2H), 2.09-2.20 (m, 1H), 3.07 (d, *J* = 5.7 Hz, 2H), 5.08 (tqq, *J* = 7.2, 1.4, 1.4 Hz, 1H), 5.45-5.50 (m, 2H).

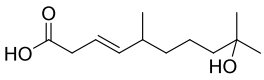
^{13}C-NMR (100.613 MHz, $CDCl_3$): δ = 17.7, 20.4, 25.8, 25.8, 36.3, 37.0, 37.8, 119.3, 124.6, 131.4, 141.2, 177.7.

Synthesis of (*E*)-9-Methoxy-5,9-dimethyl-dec-3-enoic acid



According to general procedure 1, (*E*)-9-Methoxy-5,9-dimethyl-dec-3-enoic acid (4.10 g, 90%) was obtained from commercially available 7-Methoxy-3,7-dimethyloctanal and Malonic acid. The analytical data were identical to those reported previously.[7]

Synthesis of (*E*)-9-Hydroxy-5,9-dimethyl-dec-3-enoic acid



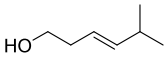
According to general procedure 1, (*E*)-9-Hydroxy-5,9-dimethyl-dec-3-enoic acid (3.64 g, 85%) was obtained from commercially available 3,7-Dimethyl-7-hydroxyoctanal and Malonic acid.

^{1}H-NMR (400.130 MHz, $CDCl_3$): δ = 0.98 (d, *J* = 6.7 Hz, 3H), 1.20 (s, 6H), 1.22-1.52 (m, 6H), 2.15 (qdt, *J* = 6.8, 6.8, 6.6 Hz, 1H), 3.05 (d, *J* = 6.1 Hz, 2H), 5.38-5.54 (m, 2H), 5.87 (s_{br}, 2H, COOH, OH).

^{13}C-NMR (100.613 MHz, $CDCl_3$): δ = 20.6, 21.9, 29.0, 29.2, 36.7, 37.2, 37.9, 43.7, 71.5, 119.7, 140.9, 177.3.

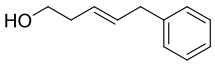
Synthesis of the substrates for hydroformylation

Synthesis of (*E*)-5-Methyl-hex-3-en-1-ol



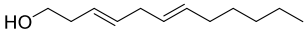
According to general procedure 2, (*E*)-5-Methyl-hex-3-en-1-ol (739 mg, 72%) was obtained by $LiAlH_4$ reduction of (*E*)-5-Methyl-hex-3-enoic acid methyl ester. The analytical data were identical to those reported previously.[8]

Synthesis of (*E*)-5-Phenyl-pent-3-en-1-ol



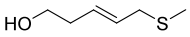
According to general procedure 2, (*E*)-5-Phenyl-pent-3-en-1-ol (1.21 g, 83%) was obtained by $LiAlH_4$ reduction of (*E*)-5-Phenyl-pent-3-enoic acid. The analytical data were identical to those reported previously.[9]

Synthesis of (*E,E*)-Dodeca-3,6-dien-1-ol



According to general procedure 2, (*E,E*)-Dodeca-3,6-dien-1-ol (1.19 g, 73%) was obtained by $LiAlH_4$ reduction of (*E,E*)-Dodeca-3,6-dienoic acid methyl ester. The analytical data were identical to those reported previously.[10]

Synthesis of (*E*)-5-Methylsulfanyl-pent-3-en-1-ol



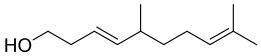
According to general procedure 2, (*E*)-5-Methylsulfanyl-pent-3-en-1-ol (939 mg, 79%) was obtained by $LiAlH_4$ reduction of (*E*)-5-Methylsulfanyl-pent-3-enoic acid.

^{1}H-NMR (400.130 MHz, $CDCl_3$): δ = 1.42 (s_{br}, 1H, OH), 2.02 (s, 3H, S-CH_3), 2.30-2.37 (m, 2H, 2-H), 3.07-3.11 (m, 2H, 5-H), 3.63-3.72 (m, 2H, 1-H), 5.47-5.60 (m, 2H, 3-H and 4-H).

^{13}C-NMR (100.613 MHz, $CDCl_3$): δ = 14.5, 35.7, 36.0, 62.1, 129.0, 129.3.

HRMS (EI): calculated: 132.060885, found: 132.061103.

Synthesis of (*E*)-5,9-Dimethyl-deca-3,8-dien-1-ol



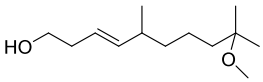
According to general procedure 2, (*E*)-5,9-Dimethyl-deca-3,8-dien-1-ol (1.39 g, 85%) was obtained by $LiAlH_4$ reduction of (*E*)-5,9-Dimethyl-deca-3,8-dienoic acid.

^{1}H-NMR (400.132 MHz, $CDCl_3$): δ = 0.97 (d, *J* = 6.7 Hz, 3H, 5-CH_3), 1.26-1.33 (m, 2H), 1.38-1.45 (m, 1H, OH), 1.59 (d, *J* = 0.9 Hz, 3H, 9-CH_3), 1.68 (d, *J* = 1.3 Hz, 3H, 10-H), 1.90-2.02 (m, 2H), 2.11 (qdt, *J* = 6.9, 6.9, 6.9 Hz, 1H, 5-H), 2.26 (dt, *J* = 6.3, 6.3 Hz, 2H), 3.62 (dt, *J* = 6.1, 6.0 Hz, 2H, 1-H), 5.09 (tqq, *J* = 7.1, 1.4, 1.4 Hz, 1H, 8-H), 5.29-5.38 (m, 1H, 3-H), 5.43 (ddt, *J* = 15.4, 7.3, 1.0 Hz, 1H, 4-H).

^{13}C-NMR (100.613 MHz, $CDCl_3$): δ = 17.8, 20.8, 25.8, 25.9, 36.1, 36.5, 37.2, 62.2, 124.1, 124.7, 131.4, 140.3.

HRMS (CI, NH_3): calculated $(M+NH_4)^+$: 200.201439, found: 200.201497.

Synthesis of (*E*)-9-Methoxy-5,9-dimethyl-dec-3-en-1-ol



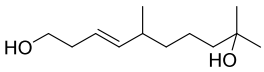
According to general procedure 2, (*E*)-9-Methoxy-5,9-dimethyl-dec-3-en-1-ol (1.52 g, 79%) was obtained by $LiAlH_4$ reduction of (*E*)-9-Methoxy-5,9-dimethyl-dec-3-enoic acid.

^{1}H-NMR (400.130 MHz, $CDCl_3$): δ = 0.96 (d, *J* = 6.7 Hz, 3H, 5-CH_3), 1.11 (s, 6H, 9-CH_3, 10-H), 1.20-1.49 (m, 6H), 1.64 (s_{br}, 1H, OH), 2.11 (dtq, *J* = 6.8, 6.7, 6.7 Hz, 1H, 5-H), 2.25 (dt, *J* = 6.4, 6.3, 2H), 3.15 (s, 3H, OCH_3), 3.60 (t, *J* = 6.3 Hz, 2H, 1-H), 5.28-5.44 (m, 2H, 3-H, 4-H).

^{13}C-NMR (100.613 MHz, $CDCl_3$): δ = 20.9, 21.6, 24.9, 25.0, 36.1, 36.8, 37.5, 40.0, 49.1, 62.2, 74.7, 124.2, 140.1.

HRMS (CI, NH_3): calculated $(M+NH_4)^+$: 232.227654, found: 232.227100.

Synthesis of (*E*)-5,9-Dimethyl-dec-3-ene-1,9-diol



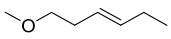
According to general procedure 2, (*E*)-5,9-Dimethyl-dec-3-ene-1,9-diol (1.44 g, 80%) was obtained by $LiAlH_4$ reduction (in this case 1.5 eq. $LiAlH_4$ was used) of (*E*)-9-Hydroxy-5,9-dimethyl-dec-3-enoic acid.

^{1}H-NMR (400.130 MHz, $CDCl_3$): δ = 0.98 (d, *J* = 6.7 Hz, 3H, 5-CH_3), 1.20 (s, 6H, 9-CH_3, 10-H), 1.23-1.51 (m, 7H, including OH), 1.67 (s_{br}, 1H, OH), 2.13 (dtq, *J* = 6.8, 6.8, 6.8 Hz, 1H, 5-H), 2.26 (td, *J* = 6.2, 6.1 Hz, 2H, 2-H), 3.62 (t, *J* = 6.3 Hz, 2H, 1-H), 5.30-5.45 (m, 2H, 3-H, 4-H).

^{13}C-NMR (100.613 MHz, $CDCl_3$): δ = 20.9, 22.0, 29.2, 29.5, 36.1, 36.8, 37.3, 43.8, 62.2, 71.1, 124.4, 140.0.

HRMS (CI, NH_3): calculated $(M+NH_4)^+$: 218.212004, found: 218.212398.

Synthesis of (*E*)-1-Methoxy-hex-3-ene



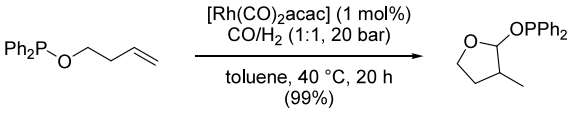
NaH (60% suspension in oil, 300 mg) was washed with THF. The remaining pure NaH (180 mg, 7.50 mmol, 1.5 eq.) was suspended in THF (6 ml). At 0 °C MeI (934 µl, 2.13 g, 15.0 mmol, 3.0 eq.) and *trans*-3-Hexen-1-ol (613 µl, 501 mg, 5.00 mmol) were added. The reaction mixture was allowed to warm up to RT and was stirred for further 16 h. The reaction was quenched with water (5 ml) and the mixture was extracted with Et_2O (3 x 20 ml). The combined organic phases were dried over $MgSO_4$ and the solvents were removed under reduced pressure. The crude product was purified by column chromatography (pentane:Et_2O = 70:30) to give the title compound (353 mg, 62 %).

^{1}H-NMR (400.130 MHz, $CDCl_3$): δ = 0.97 (t, *J* = 7.5 Hz, 3H), 2.02 (m_c, 2H), 2.27 (m_c, 2H), 3.34 (s, 3H), 3.39 (t, *J* = 6.8 Hz, 2H), 5.35-5.45 (m, 1H), 5.50-5.59 (m, 1H).

^{13}C-NMR (100.613 MHz, $CDCl_3$): δ = 13.8, 25.7, 33.0, 58.7, 72.8, 125.3, 134.3.

Hydroformylation reactions

Hydroformylation of Homoallyl-diphenylphosphinite (2)



Homoallyl-diphenylphosphinite (**2**) (174 mg, 0.680 mmol) was dissolved in toluene (3.4 ml). $Rh(CO)_2acac$ (1.8 mg, 0.0068 mmol) was added and the reaction mixture was transferred to the parallel autoclave. The reaction was heated to 40 °C and pressurized with CO/H_2 (1:1, 20 bar). The reaction mixture was stirred under these conditions for 20 h. After the pressure was released, the reaction mixture was filtrated over Alox N. The solvent was evaporated to yield the title compound (192 mg, 99%). One part of the product was analyzed by NMR. The other part was dissolved in MeOH (3 ml) and Tetrazole (5 mg) was added to liberate the free lactol. This sample was analyzed by GC. Method: 135 °C, hold 10 min.; 20 °C/min. to 250 °C, hold 15 min. Retention times: γ-lactol: 5.03 min, δ-lactol: 6.72 min.
Regioselectivity: 99 : 1.

NMR-analysis of the lactol phosphinites (2 diastereomers) [signals of the other diastereomer, if separated]:
^{1}H-NMR (400.130 MHz, C_6D_6): δ = 0.67 (d, *J* = 7.2 Hz, 3H), [0.97 (d, *J* = 6.7 Hz)], 1.01-2.32 (m, 3H), 3.49-3.96 (m, 2H), 4.90-5.66 (m, 1H), 7.00-7.18 (m, 6H), 7.56-7.70 (m, 4H).

^{13}C-NMR (100.613 MHz, C_6D_6) [doubled signal set due to 2 diastereomers]: δ = 13.3, 17.1, 30.8, 31.2, 40.1 (d, *J* = 6.3 Hz), 41.4 (d, *J* = 6.0 Hz), 67.3, 67.6, 106.2 (d, *J* = 18.1 Hz), 110.6 (d, *J* = 18.6 Hz), 128.4 (d, *J* = 6.5 Hz, 2C), 128.4 (d, *J* = 7.2 Hz, 2C), 128.5 (d, *J* = 7.0 Hz, 2C), 128.6 (d, *J* = 11.1 Hz, 2C), 129.1 (2C), 129.2, 129.2, 130.7 (d, *J* = 21.7 Hz, 2C), 130.8 (d, *J* = 21.5 Hz, 2C), 130.9 (d, *J* = 22.2 Hz), 131.0 (d, *J* = 22.2 Hz), 131.0 (d, *J* = 22.0 Hz, 2C), 143.1 (d, *J* = 15.5 Hz), 143.2 (d, *J* = 15.0 Hz), 143.6 (d, *J* = 19.3 Hz), 144.0 (d, *J* = 20.0 Hz).

^{31}P-NMR (121.468 MHz, C_6D_6): δ = 104.1, 104.9.

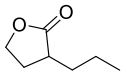
Synthesis of 3-Methyl-dihydro-furan-2-one

Following general procedure 3, 3-Methyl-dihydro-furan-2-one (54.6 mg, 91%) was obtained on hydroformylation of But-3-ene-1-ol and PCC-oxidation of the lactol.

GC-analysis for the determination of conversion and regioselectivity: Method: 135 °C, hold 10 min.; 20 °C/min. to 250 °C, hold 15 min. Retention times: But-3-en-1-ol: 2.53 min., γ-lactol: 5.03 min, δ-lactol: 6.72 min.
Conversion: 100%, Regioselectivity: 99 : 1.

The analytical data of the title compound were identical to those reported previously.[11]

Synthesis of 3-Propyl-dihydro-furan-2-one from *trans*-Hex-3-ene-1-ol

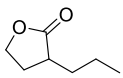


Following general procedure 3, 3-Propyl-dihydro-furan-2-one (65.3 mg, 85%) was obtained on hydroformylation of *trans*-Hex-3-ene-1-ol and PCC-oxidation of the lactol.

GC-analysis for the determination of conversion and regioselectivity: Method: 155 °C, hold 10 min.; 20 °C/min. to 250 °C, hold 15 min. Retention times: *trans*-Hex-3-ene-1-ol: 2.68 min., γ-lactol: 5.55 min., δ-lactol: 6.08 min.
Conversion: 100%, Regioselectivity: >99 : <1.

The analytical data of the title compound were identical to those reported previously.[12]

Synthesis of 3-Propyl-dihydro-furan-2-one from *cis*-Hex-3-ene-1-ol

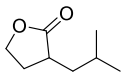


Following general procedure 3, 3-Propyl-dihydro-furan-2-one (67.6 mg, 88%) was obtained on hydroformylation of *cis*-Hex-3-ene-1-ol and PCC-oxidation of the lactol.

GC-analysis for the determination of conversion and regioselectivity: Method: 155 °C, hold 10 min.; 20 °C/min. to 250 °C, hold 15 min. Retention times: *cis*-Hex-3-ene-1-ol: 2.77 min., γ-lactol: 5.55 min., δ-lactol: 6.08 min.
Conversion: 100%, Regioselectivity: >99 : <1.

The analytical data of the title compound were identical to those reported previously.[12]

Synthesis of 3-Isobutyl-dihydro-furan-2-one

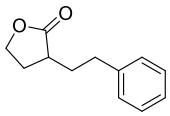


Following general procedure 3, 3-Isobutyl-dihydro-furan-2-one (70.1 mg, 82%, 84% based on conversion) was obtained on hydroformylation of (*E*)-5-Methyl-hex-3-en-1-ol and PCC-oxidation of the lactol.

GC-analysis for the determination of conversion and regioselectivity: Method: 135 °C, hold 10 min.; 20 °C/min. to 250 °C, hold 15 min. Retention times: (*E*)-5-Methyl-hex-3-en-1-ol: 3.57 min., γ-lactol: 10.73 min., δ-lactol: 12.03 min.
Conversion: 98%, Regioselectivity: 97 : 3.

The analytical data of the title compound were identical to those reported previously.[13]

Synthesis of 3-Phenethyl-dihydro-furan-2-one

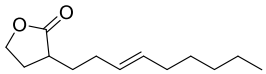


Following general procedure 3, 3-Phenethyl-dihydro-furan-2-one (87.8 mg, 77 %, 92% based on conversion) was obtained on hydroformylation of (*E*)-5-Phenyl-pent-3-en-1-ol and PCC-oxidation of the lactol.

GC-analysis for the determination of conversion and regioselectivity: Method: 200 °C, hold 10 min.; 20 °C/min. to 250 °C, hold 15 min. Retention times: (*E*)-5-Phenyl-pent-3-en-1-ol: 7.31 min., γ-lactols (the 2 diastereomers are separated): 14.22, 15.09 min., δ-lactols (the 2 diastereomers are separated): 14.02, 15.45 min.
Conversion: 84%, Regioselectivity: 99 : 1.

The analytical data of the title compound were identical to those of the literature.[14]

Synthesis of 3-Non-3-enyl-dihydro-furan-2-one



Following general procedure 3, 3-Non-3-enyl-dihydro-furan-2-one (84.4 mg, 67%, 83% based on conversion) was obtained on hydroformylation of (*E,E*)-Dodeca-3,6-dien-1-ol and PCC-oxidation of the lactol.

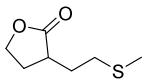
GC-analysis for the determination of conversion and regioselectivity: Method: 200 °C, hold 10 min.; 20 °C/min. to 250 °C, hold 15 min. Retention times: (*E,E*)-Dodeca-3,6-dien-1-ol: 4.08 min., γ-lactols (the 2 diastereomers are separated): 10.20, 11.38 min., other products including double hydroformylated products: 10.44, 11.17, 11.49 min.
Conversion: 81%, Regioselectivity (γ-lactol vs. all other products): 97 : 3.

^{1}H-NMR (400.130 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 7.0 Hz, 3H), 1.20-1.42 (m, 6H), 1.44-1.56 (m, 1H), 1.86-2.24 (m, 6H), 2.34-2.45 (m, 1H), 2.48-2.59 (m, 1H), 4.18 (ddd, *J* = 9.4, 9.3, 6.7 Hz, 1H), 4.34 (ddd, *J* = 8.8, 8.8, 2.9 Hz, 1H), 5.30-5.52 (m, 2H).

^{13}C-NMR (100.613 MHz, $CDCl_3$): δ = 14.1, 22.6, 28.7, 29.2, 30.3 (2C), 31.5, 32.6, 38.6, 66.5, 128.4, 132.2, 179.6.

HRMS (EI): calculated: 210.161980, found: 210.162005

Synthesis of 3-(2-Methylsulfanyl-ethyl)-dihydro-furan-2-one



Following general procedure 3, 3-(2-Methylsulfanyl-ethyl)-dihydro-furan-2-one (30.3 mg, 32%, 42% based on conversion) was obtained on hydroformylation of (*E*)-5-Methylsulfanyl-pent-3-en-1-ol and PCC-oxidation of the lactol.

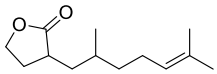
GC-analysis for the determination of conversion and regioselectivity: Method: 155 °C, hold 10 min.; 20 °C/min. to 250 °C, hold 15 min. Retention times: (*E*)-5-Methylsulfanyl-pent-3-en-1-ol: 11.00 min., γ-lactols (the 2 diastereomers are separated): 15.33, 15.62 min., δ-lactols (the 2 diastereomers are separated): 15.15, 16.28 min.
Conversion: 75%, Regioselectivity: 97 : 3.

^{1}H-NMR (400.130 MHz, $CDCl_3$): δ = 1.67 (dddd, *J* = 14.1, 8.6, 7.7, 5.8 Hz, 1H), 1.89 (dddd, *J* = 12.5, 10.4, 10.3, 8.7 Hz, 1H), 2.05 (s, 3H, SCH_3), 2.06-2.18 (m, 1H), 2.37 (dddd, *J* = 12.6, 8.8, 6.5, 2.5 Hz, 1H), 2.52 (ddd, *J* = 13.3, 7.6, 7.6 Hz, 1H), 2.62 (ddd, *J* = 13.3, 7.8, 5.7 Hz, 1H), 2.70 (dddd, *J* = 10.7, 8.7, 8.7, 5.3 Hz, 1H), 4.14 (ddd, *J* = 10.0, 9.1, 6.7 Hz, 1H), 4.30 (ddd, *J* = 8.8, 8.8, 2.6 Hz, 1H).

^{13}C-NMR (100.613 MHz, $CDCl_3$): δ = 14.2, 27.7, 28.5, 30.7, 36.9, 65.4, 178.1.

HRMS (CI, NH_3): calculated $(M+H)^+$: 161.063625, found: 161.063404.

Synthesis of 3-(2,6-Dimethyl-hept-5-enyl)-dihydro-furan-2-one



Following general procedure 3, 3-(2,6-Dimethyl-hept-5-enyl)-dihydro-furan-2-one (116 mg, 92%, 99% based on conversion) was obtained on hydroformylation of (*E*)-5,9-Dimethyl-deca-3,8-dien-1-ol and PCC-oxidation of the lactol.

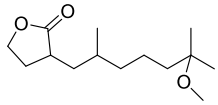
GC-analysis for the determination of conversion: Method: 200 °C, hold 10 min.; 20 °C/min. to 250 °C, hold 15 min. Retention times: (*E*)-5,9-Dimethyl-deca-3,8-dien-1-ol: 3.46 min., γ-lactols (3 peaks for 4 diastereomers): 8.54, 9.96, 10.20 min., δ-lactols not detected due to low conversion of PPh_3 hydroformylation. Determination of regioselctivity by ^{1}H- and ^{13}C-NMR analysis after oxidation to the corresponding lactones, where no δ-lactone was detected.
Conversion: 93%, Regioselectivity: 99 : 1.

^{1}H-NMR (400.130 MHz, $CDCl_3$) [signals of the other diastereomer, if separated]: δ = 0.92 (d, *J* = 6.6 Hz, 3H), [0.95 (d, *J* = 6.7 Hz], 1.08-1.57 (m, 4H), 1.60 (s, 3H), 1.68 (s, 3H), 1.70-2.10 (m, 4H), 2.34-2.46 (m, 1H), 2.51-2.64 (m, 1H), 4.14-4.23 (m, 1H), 4.31-4.39 (m, 1H), 5.08 (ddqq, *J* = 8.5, 5.7, 1.4, 1.4 Hz, 1H).

^{13}C-NMR (100.613 MHz, $CDCl_3$) [doubled signal set due to 2 diastereomers]: 17.7 (2C), 18.6, 20.0, 25.3, 25.5, 25.8 (2C), 29.1, 29.6, 30.6, 30.7, 36.0, 37.4 (2C), 37.5, 37.6, 38.1, 66.4, 66.5, 124.5, 124.5, 131.5, 131.6, 179.9, 180.1.

HRMS (EI): calculated: 210.161980, found: 210.161499.

Synthesis of 3-(6-Methoxy-2,6-dimethyl-heptyl)-dihydro-furan-2-one



Following general procedure 3, 3-(6-Methoxy-2,6-dimethyl-heptyl)-dihydro-furan-2-one (126 mg, 86%, 88% based on conversion) was obtained on hydroformylation of (*E*)-9-Methoxy-5,9-dimethyl-dec-3-en-1-ol and PCC-oxidation of the lactol.

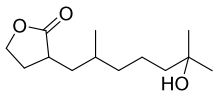
GC-analysis for the determination of conversion: Method: 200 °C, hold 10 min.; 20 °C/min. to 250 °C, hold 15 min. Retention times: (*E*)-9-Methoxy-5,9-dimethyl-dec-3-en-1-ol: 5.06 min., γ-lactols (3 peaks for 4 diastereomers): 12.58, 13.30, 13.43 min., δ-lactols not detected due to low conversion of PPh_3 hydroformylation. Determination of regioselctivity by ^{1}H- and ^{13}C-NMR analysis after oxidation to the corresponding lactones, where no δ-lactone was detected.
Conversion: 98%, Regioselectivity: 99 : 1.

^{1}H-NMR (400.130 MHz, $CDCl_3$) [signals of the other diastereomer, if separated]: δ = 0.85 (d, *J* = 6.6 Hz, 3H), [0.88 (d, *J* = 6.6 Hz)], 1.07 (s, 6H), [1.07 (s)], 1.10-1.52 (m, 8H), 1.60-1.69 (m, 1H), 1.76-1.92 (m, 1H), 2.27-2.39 (m, 1H), 2.42-2.58 (m, 1H), 3.10 (s, 3H), [3.10 (s)], 4.08-4.16 (m, 1H), 4.24-4.32 (m, 1H).

^{13}C-NMR (100.613 MHz, $CDCl_3$) [doubled signal set due to 2 diastereomers]: δ = 17.6, 18.9, 20.0, 20.2, 23.9 (2C), 23.9 (2C), 28.0, 28.6, 30.0, 30.0, 35.6, 36.3 (2C), 36.7, 37.0, 37.1, 39.1, 39.3, 48.1 (2C), 65.4, 65.4, 73.5 (2C), 178.8, 179.0.

HRMS (CI, NH_3): calculated $(M+H)^+$: 243.196020, found: 243.196102.

Synthesis of 3-(6-Hydroxy-2,6-dimethyl-heptyl)-dihydro-furan-2-one



Following general procedure 3, 3-(6-Hydroxy-2,6-dimethyl-heptyl)-dihydro-furan-2-one (115 mg, 84%, 99% based on conversion) was obtained on hydroformylation of (*E*)-5,9-Dimethyl-dec-3-ene-1,9-diol and PCC-oxidation of the lactol.

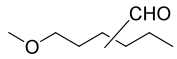
GC-analysis for the determination of conversion: Method: 200 °C, hold 10 min.; 20 °C/min. to 250 °C, hold 15 min. Retention times: (*E*)-5,9-Dimethyl-dec-3-ene-1,9-diol: 8.16 min., γ-lactols (3 peaks for 4 diastereomers): 15.33, 16.41, 16.62 min., δ-lactols not detected due to low conversion of PPh_3 hydroformylation. Determination of regioselctivity by ^{1}H- and ^{13}C-NMR analysis after oxidation to the corresponding lactones, where no δ-lactone was detected. Conversion: 85%, Regioselectivity: 99 : 1.

^{1}H-NMR (400.130 MHz, $CDCl_3$) [signals of the other diastereomer, if separated]: δ = 0.92 (d, *J* = 6.4 Hz, 3H), [0.95 (d, *J* = 6.7 Hz)], 1.21 (s, 6H), 1.22-1.79 (m, 10H), 1.82-1.99 (m, 1H), 2.34-2.47 (m, 1H), 2.51-2.65 (m, 1H), 4.14-4.24 (m, 1H), 4.31-4.39 (m, 1H).

^{13}C-NMR (100.613 MHz, $CDCl_3$) [doubled signal set due to 2 diastereomers]: δ = 18.7, 20.0, 21.5, 21.7, 29.1, 29.3, 29.4, 29.4, 29.4, 29.7, 31.0, 31.0, 36.6, 37.3 (2C), 37.7, 38.0, 38.2, 44.1, 44.1, 66.5, 66.5, 71.1 (2C), 179.9, 180.1.

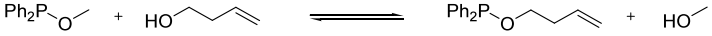
HRMS (CI): calculated (M-CH_3): 213.149070, found: 213.148997.

Hydroformylation of (*E*)-1-Methoxy-hex-3-ene



Following general procedure 3, (*E*)-1-Methoxy-hex-3-ene was hydroformylated and conversion and regioselectivity were determined by GC-analysis. Method: 135 °C, hold 10 min. Retention times: (*E*)-1-Methoxy-hex-3-ene: 2.25 min, hydroformylated products: 3.88, 4.14 min. Conversion: 6%, Regioselectivity: 53 : 47.

Transesterification experiments



Three different experiments were launched to determine the transesterification catalyst. Three flasks were charged each with Methyl-diphenylphosphinite (**3**) (108 mg, 0.50 mmol) in THF (25 ml). To each reaction mixture was added But-3-en-1-ol (434 µl, 361 mg, 5.00 mmol). To flask 1 $Rh(CO)_2acac$ (12.9 mg, 0.05 mmol) was added. To flask 2 molecular sieves (4 Å) were added. To flask 3 $Rh(CO)_2acac$ (12.9 mg, 0.05 mmol) and molecular sieves (4 Å) were added. The reaction mixtures were heated at 40 °C for 2 ½ h. After cooling to RT the reaction mixtures containing $Rh(CO)_2acac$ were filtrated over Al_2O_3 (neutral) to remove the Rhodium catalyst. The solvents of all reaction mixtures were evaporated and the residue was

analyzed by NMR spectroscopy. The samples (1 and 3) which contained $Rh(CO)_2acac$ showed both phosphinites [Homoallyl-diphenylphosphinite (**2**) as well as Methyl-diphenylphosphinite (**3**)] in a ratio of about 1 : 1. Sample 2 which contained only the molecular sieves showed only the Methyl-diphenylphosphinite (**3**) as the starting material.

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cMi06-4010,Gruenanger,cg-273,CDCl3

BRUKER

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PROCNO                       1

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DW                     60.400 usec
DE                      6.00 usec
TE                     300.0 K
D1                0.10000000 sec
TD0                         1

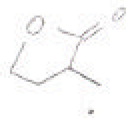
======== CHANNEL f1 ========
NUC1                       1H
P1                       9.90 usec
PL1                     -3.00 dB
SFO1          400.1324710 MHz

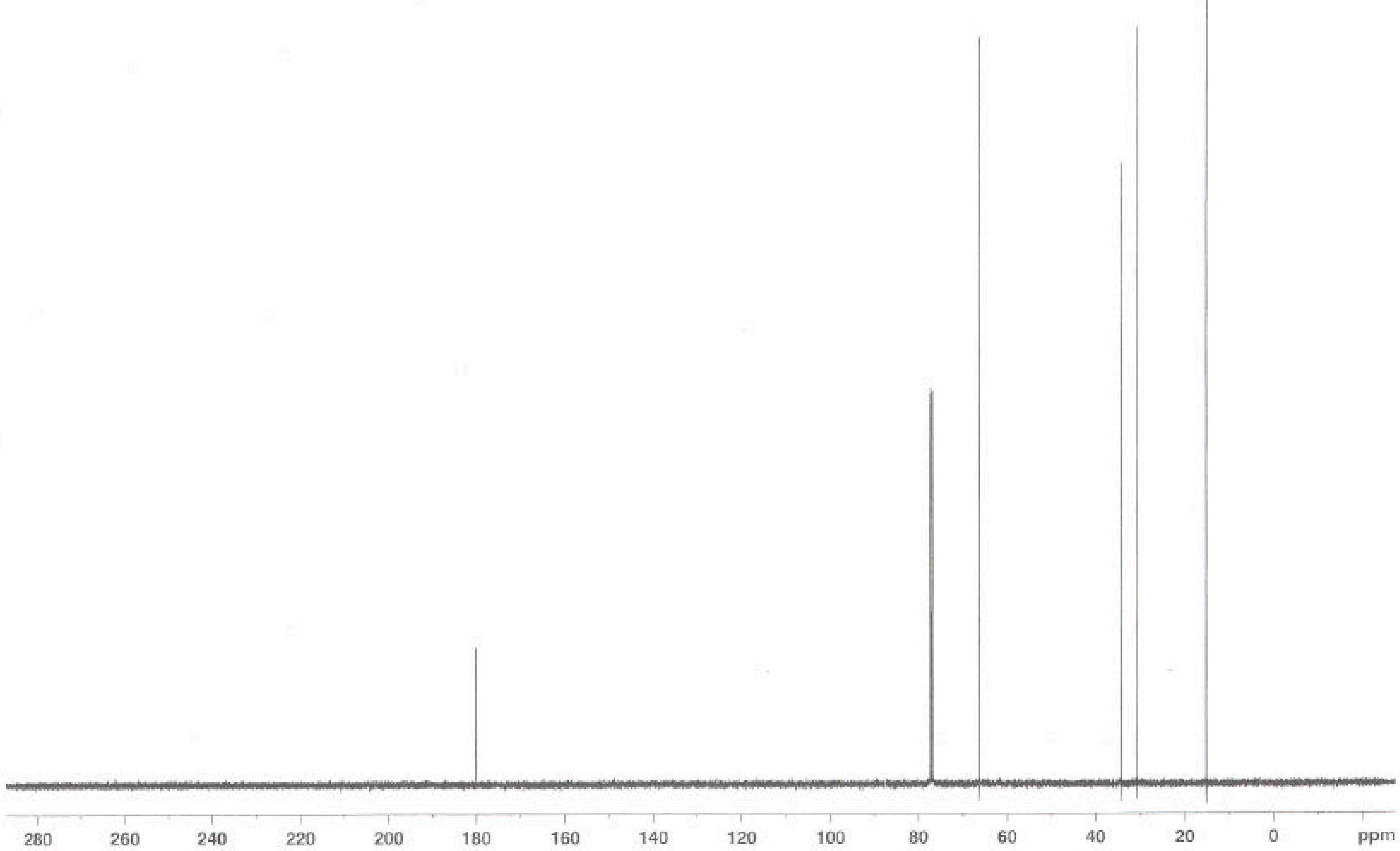
F2 - Processing parameters
SI                     65536
SF                400.1300049 MHz
WDW                       EM
SSB                        0
LB                     -0.30 Hz
GB                        0.3
PC                       1.00
SR                       4.90 Hz
```

Ai06-4011,Gruenanger,cg-273,CDCl3

#/opt/topspin, USER=[illegible], NAME=20080506B, EXPNO=11, PROCNO=1
F1=287.298ppm, F2=-17.229ppm, MI=0.18cm, MAXI=10000.00cm, PC=1.400

#	ADDRESS	FREQUENCY [Hz]	(PPM)	INTENSITY
1	44691.1	18225.588	180.1919	1.34
2	67462.8	7789.253	77.4181	6.78
3	67513.9	7777.168	77.2980	9.28
4	67595.4	7761.268	77.1000	6.85
5	67727.9	7726.268	76.7828	9.76
6	92110.8	6667.073	66.2647	13.90
7	105488.0	3437.526	34.1839	10.67
8	104818.3	3092.015	30.7318	11.08
9	113591.3	1529.164	15.1985	15.00





BRUKER

Current Data Parameters
NAME 20080506B
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080506
Time 7.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 809
DS 2
SWH 31645.570 Hz
FIDRES 0.482873 Hz
AQ 1.0355188 sec
RG 2580.3
DW 15.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.90 usec
PL1 -3.00 dB
SFO1 100.6258487 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 131072
SF 100.6127655 MHz
WDW GM
SSB 0
LB -0.50 Hz
GB 0.1
PC 1.40
SR -3.53 Hz

GrBtMi07-4010,Gruenanger,cg-275,CDCl3,TMS

BRUKER

```
Current Data Parameters
NAME                  20080507b
EXPNO                        10
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20080507
Time                      7.39
INSTRUM                   spect
PROBHD     5 mm PABBO BB-
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          64
DS                           2
SWH                 8278.146 Hz
FIDRES              0.126314 Hz
AQ                  3.9584243 sec
RG                       80.6
DW                     60.400 usec
DE                      6.00 usec
TE                     300.0 K
D1                0.10000000 sec
TD0                          1

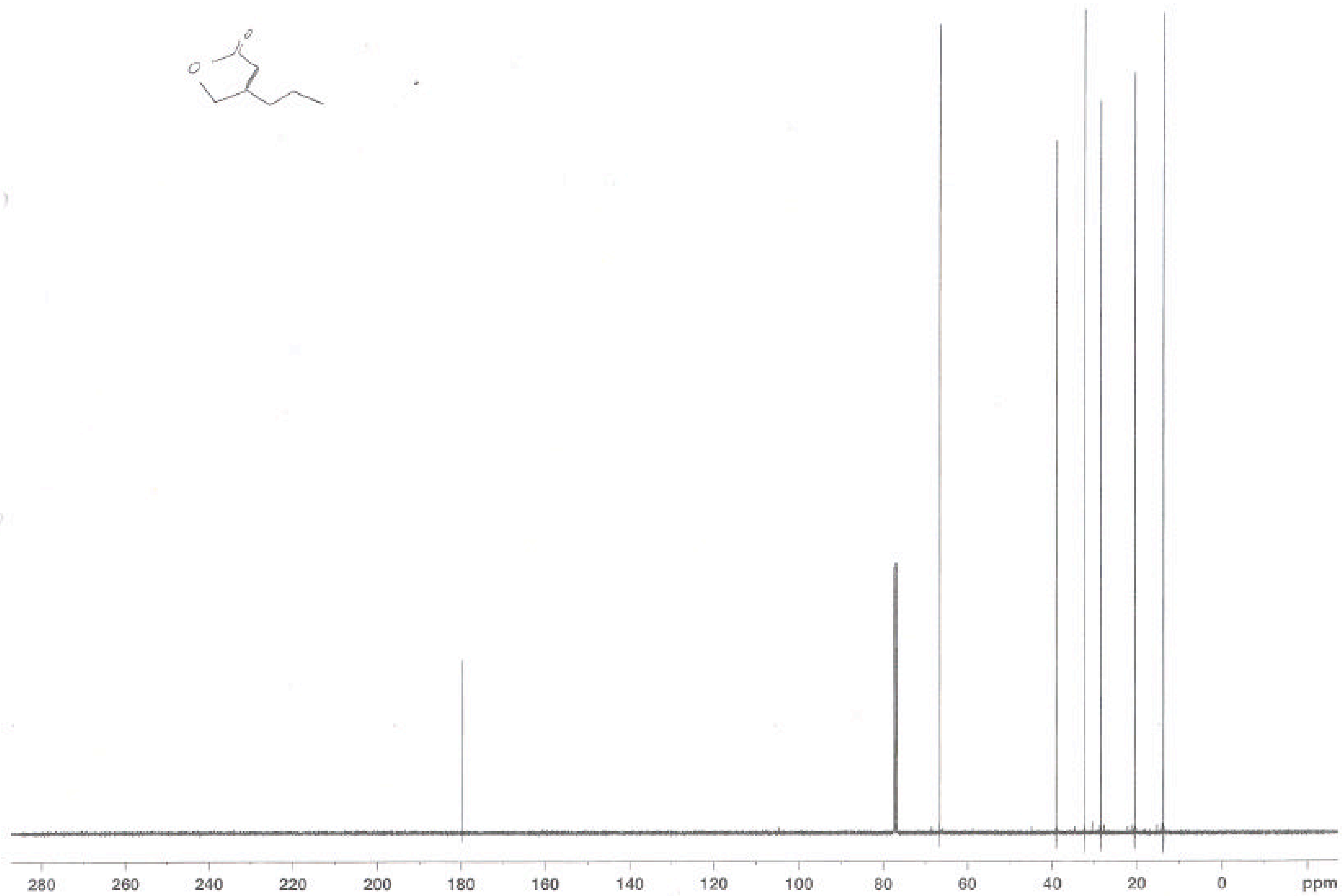
======== CHANNEL f1 ========
NUC1                       1H
P1                       9.90 usec
PL1                     -3.00 dB
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                       65536
SF             400.1299888 MHz
WDW                        EM
SSB                         0
LB                      -0.30 Hz
GB                         0.3
PC                        1.00
SR                     -31.18 Hz
```

rBtMi07-4011,Gruenanger,cg-275,CDCl3,TMS

/u/opt/topspin, USER=[illegible], NAME=200805078, EXPNO=11, PROCNO=1
F1=287.285ppm, F2=-27.244ppm, MI=1.20cm, MAXI=10000.00cm, PC=1.400

#	ADDRESS	FREQUENCY [HZ]	FREQUENCY [PPM]	INTENSITY
1	44855.4	18072.950	179.6228	3.20
2	87456.7	7785.256	77.6282	4.85
3	87505.7	7777.433	77.5887	0.25
4	87589.3	7757.243	77.1080	4.90
5	87721.9	7725.223	76.7817	4.92
6	92008.3	6690.341	66.4958	34.06
7	103469.9	3923.091	38.9920	32.63
8	106215.0	3260.939	32.4047	36.00
9	107018.3	3066.421	30.4776	0.29
10	107799.8	2877.658	28.6013	33.35
11	111172.8	2063.323	20.5076	33.91
12	113902.2	1385.340	13.7663	34.97



BRUKER

Current Data Parameters
NAME 20080507B
EXPNO 11
PROCNO 1

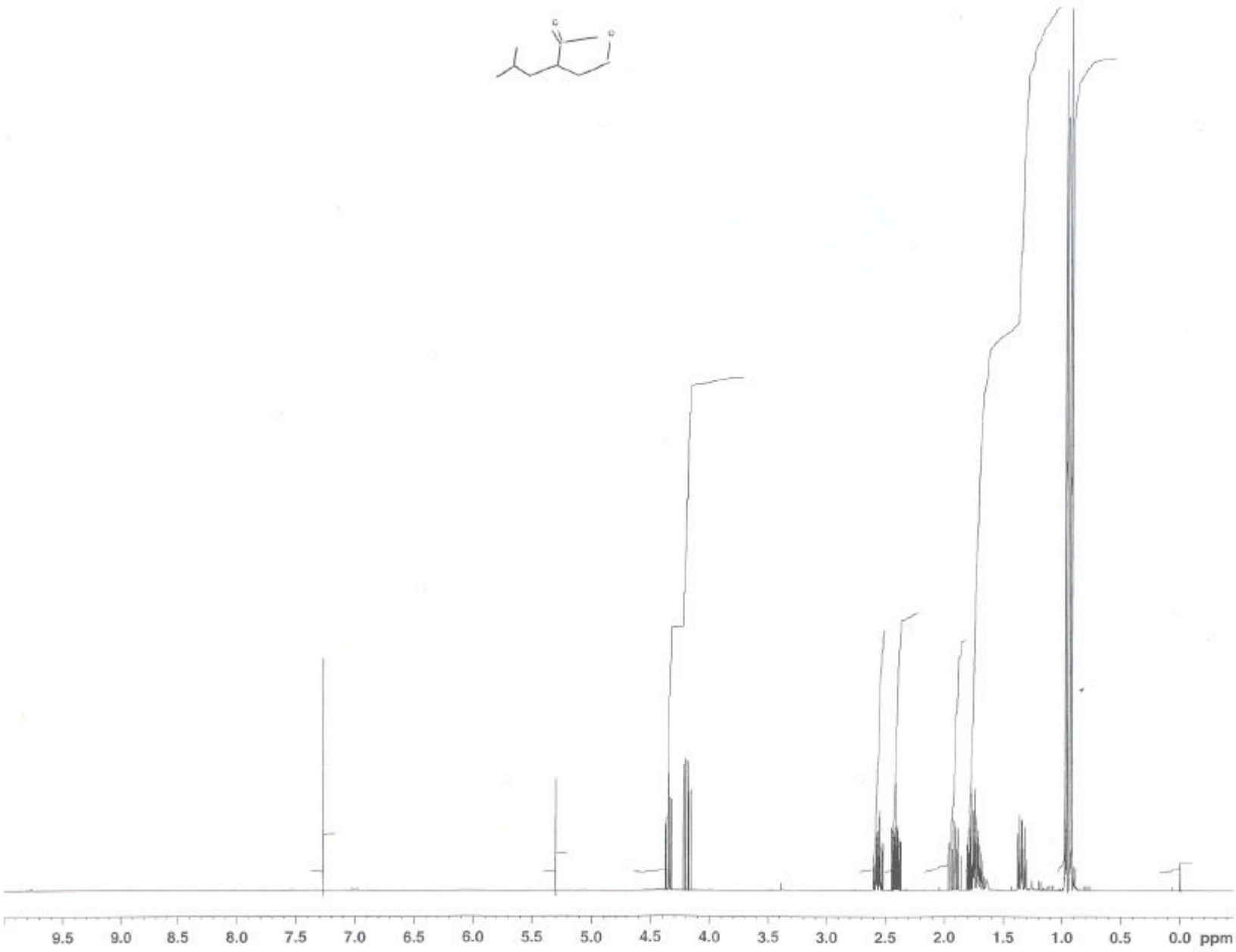
F2 - Acquisition Parameters
Date_ 20080507
Time 8.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1400
DS 2
SWH 31645.570 Hz
FIDRES 0.482873 Hz
AQ 1.0355188 sec
RG 14596.5
DW 15.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.90 usec
PL1 -3.00 dB
SFO1 100.6258487 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 131072
SF 100.6127669 MHz
WDW EM
SSB 0
LB -0.50 Hz
GB 0.1
PC 1.40
SR -2.05 Hz

GrEtM107-4060,Gruenanger,cg-276-2,CDCl3,TM



BRUKER

```
Current Data Parameters
NAME                  20080507B
EXPNO                        60
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20080507
Time                      15.05
INSTRUM                   spect
PROBHD     5 mm PABBO BB-
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          64
DS                           2
SWH                   8278.146 Hz
FIDRES                0.126314 Hz
AQ                    3.9584243 sec
RG                        203.2
DW                       60.400 usec
DE                        6.00 usec
TE                       300.0 K
D1                    0.10000000 sec
TD0                           1

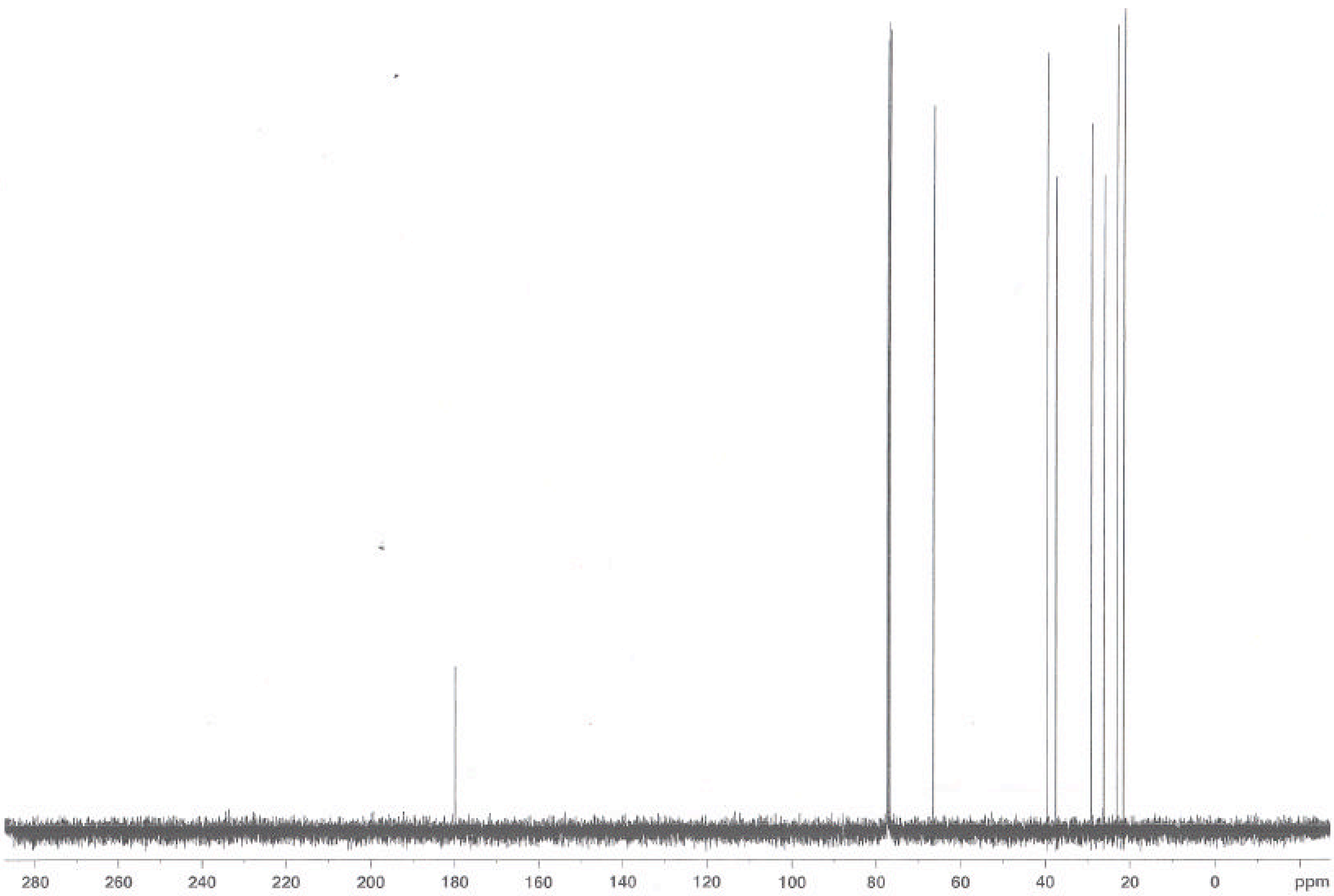
======== CHANNEL f1 ========
NUC1                         1H
P1                         9.90 usec
PL1                       -3.00 dB
SFO1            400.1324710 MHz

F2 - Processing parameters
SI                       65536
SF              400.1300001 MHz
WDW                         EM
SSB                          0
LB                       -0.30 Hz
GB                         0.3
PC                        1.00
SR                        0.09 Hz
```

1107-4062,Gruenanger,cg-276-2,CDCl3,TMS

[illegible]

#	ADDRESS	FREQUENCY [Hz]	FREQUENCY [PPM]	INTENSITY
1	44763.1	18107.566	179.9729	7.57
2	87480.5	7789.197	77.4176	16.36
3	87528.1	7777.650	77.3026	0.61
4	87612.9	7757.241	77.1006	16.87
5	87745.3	7725.264	76.7823	16.49
6	92032.9	6690.098	66.4935	15.10
7	103227.6	3987.272	39.6295	14.20
8	104086.5	3780.365	37.5736	12.04
9	107545.4	2945.252	29.2735	12.89
10	108794.8	2643.882	26.2776	11.83
11	110085.3	2331.558	23.1737	14.67
12	110777.6	2164.426	21.5124	15.00



BRUKER

```
Current Data Parameters
NAME          20080507D
EXPNO                62
PROCNO                1

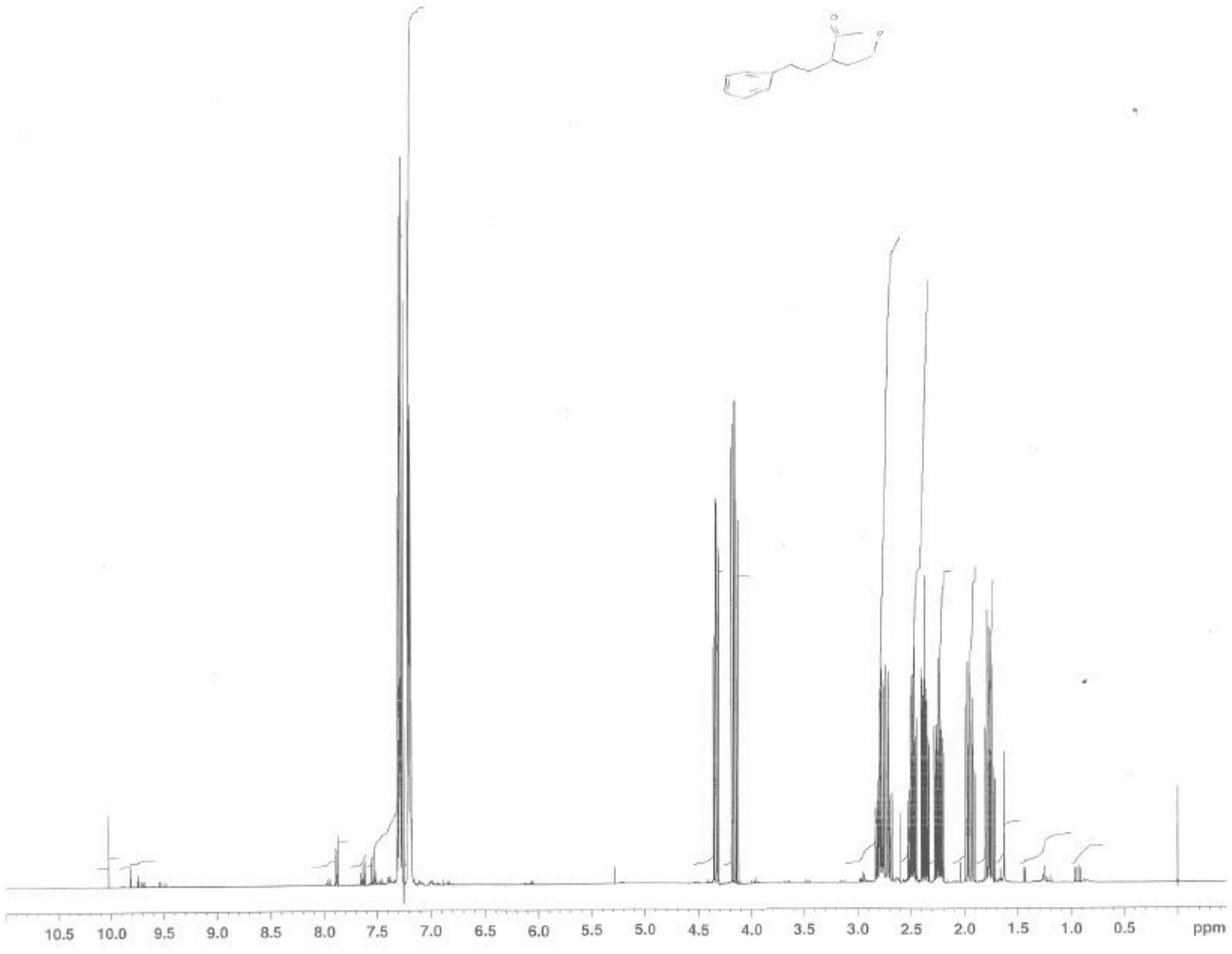
F2 - Acquisition Parameters
Date_          20080507
Time              15.29
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  320
DS                    2
SWH           31645.570 Hz
FIDRES         0.482873 Hz
AQ            1.0355188 sec
RG                645.1
DW               15.800 usec
DE                 6.00 usec
TE                300.0 K
D1           1.00000000 sec
d11          0.03000000 sec
DELTA        0.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 7.90 usec
PL1               -3.00 dB
SFO1        100.6258487 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -3.00 dB
PL12              15.00 dB
PL13              15.00 dB
SFO2        400.1320007 MHz

F2 - Processing parameters
SI               131072
SF          100.6127613 MHz
WDW                  EM
SSB                   0
LB                -0.50 Hz
GB                  0.1
PC                 1.40
SR                -7.75 Hz
```

GrBtM107-4070,Gruenanger,cg-276-5,CDCl3,TM




```
Current Data Parameters
NAME                  20080507b
EXPNO                        70
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20080507
Time                      21.55
INSTRUM                   spect
PROBHD    5 mm PABBO BB-
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          64
DS                           2
SWH                   8278.146 Hz
FIDRES                0.126314 Hz
AQ                    3.9584243 sec
RG                         161
DW                      60.400 usec
DE                       6.00 usec
TE                      300.0 K
D1                    0.10000000 sec
TD0                           1

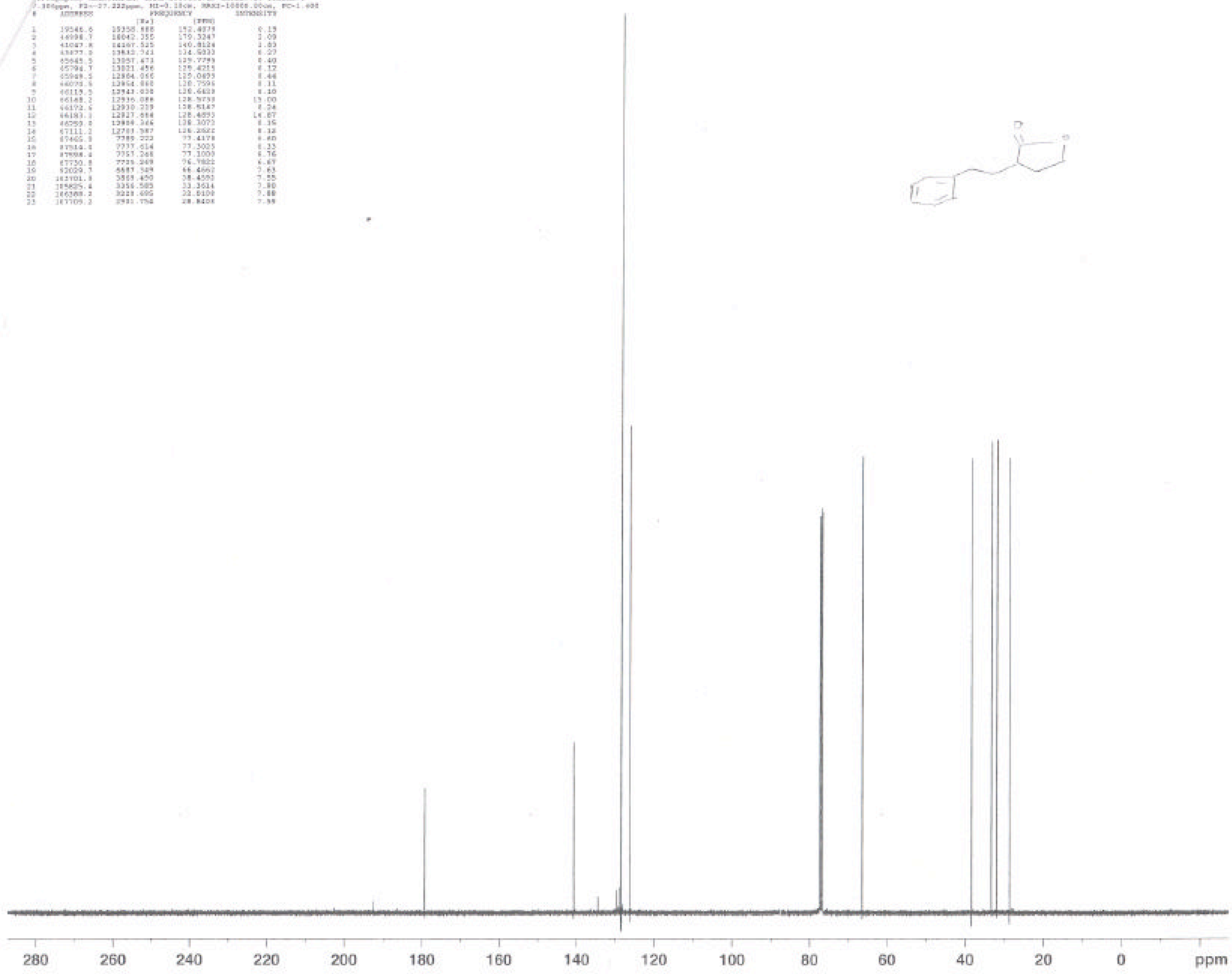
======== CHANNEL f1 ========
NUC1                         1H
P1                        9.90 usec
PL1                      -3.00 dB
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                       65536
SF                  400.1300079 MHz
WDW                         GM
SSB                          0
LB                       -0.30 Hz
GB                         0.3
PC                        1.00
SR                        7.93 Hz
```

107-407B, Gruenanger, cg-276-5, CDCl3, TMS

F:/topspin, USERname, NAME=20080507B, EXPNO=72, PROCNO=1
F1=...ppm, F2=-27.222ppm, MI=0.10cm, MAXI=10000.00cm, PC=1.400

#	ADDRESS	FREQUENCY [Hz]	FREQUENCY [PPM]	INTENSITY
1	19546.6	19358.888	192.4879	0.15
2	44998.7	18042.355	179.3247	2.09
3	41047.8	14167.525	140.8124	3.83
4	43877.3	13542.741	134.5333	0.27
5	65645.5	13057.673	129.7799	0.40
6	65794.7	13021.456	129.4215	0.12
7	65949.3	12984.065	129.0875	0.46
8	66075.5	12954.860	128.7596	0.11
9	66119.5	12943.030	128.6620	0.10
10	66148.2	12936.088	128.5750	13.00
11	66172.6	12930.229	128.5147	0.24
12	66183.1	12927.888	128.4893	14.87
13	66250.0	12908.366	128.3072	0.16
14	67111.2	12703.587	126.2622	0.12
15	87465.3	7789.222	77.4178	6.60
16	87514.0	7777.614	77.3025	0.33
17	87598.4	7757.248	77.1000	6.76
18	87730.8	7725.269	76.7822	6.67
19	92029.7	6687.529	66.4662	7.63
20	102701.3	3869.450	38.4592	7.55
21	105825.4	3356.585	33.2614	7.80
22	106388.2	3221.695	32.0109	7.88
23	107109.2	2901.758	28.8408	7.99



Current Data Parameters
NAME 20080507B
EXPNO 72
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080507
Time 23.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2000
DS 2
SWH 31645.570 Hz
FIDRES 0.482873 Hz
AQ 1.0355188 sec
RG 1024
DW 15.800 usec
DE 6.00 usec
TE 299.8 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.90 usec
PL1 -3.00 dB
SFO1 100.6258487 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 131072
SF 100.6127648 MHz
WDW GM
SSB 0
LB -0.50 Hz
GB 0.1
PC 1.40
SR -4.25 Hz

GrB:Mi08-4060,Gruenanger,cg-277-8,CDCl3,TM

BRUKER

```
Current Data Parameters
NAME                 20080508B
EXPNO                       60
PROCNO                       1

F2 - Acquisition Parameters
Date_              20080508
Time                    14.30
INSTRUM                 spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        64
DS                         2
SWH                 8278.146 Hz
FIDRES              0.126314 Hz
AQ                  3.9584243 sec
RG                      161.3
DW                     60.400 usec
DE                      6.00 usec
TE                     300.0 K
D1                0.10000000 sec
TD0                          1

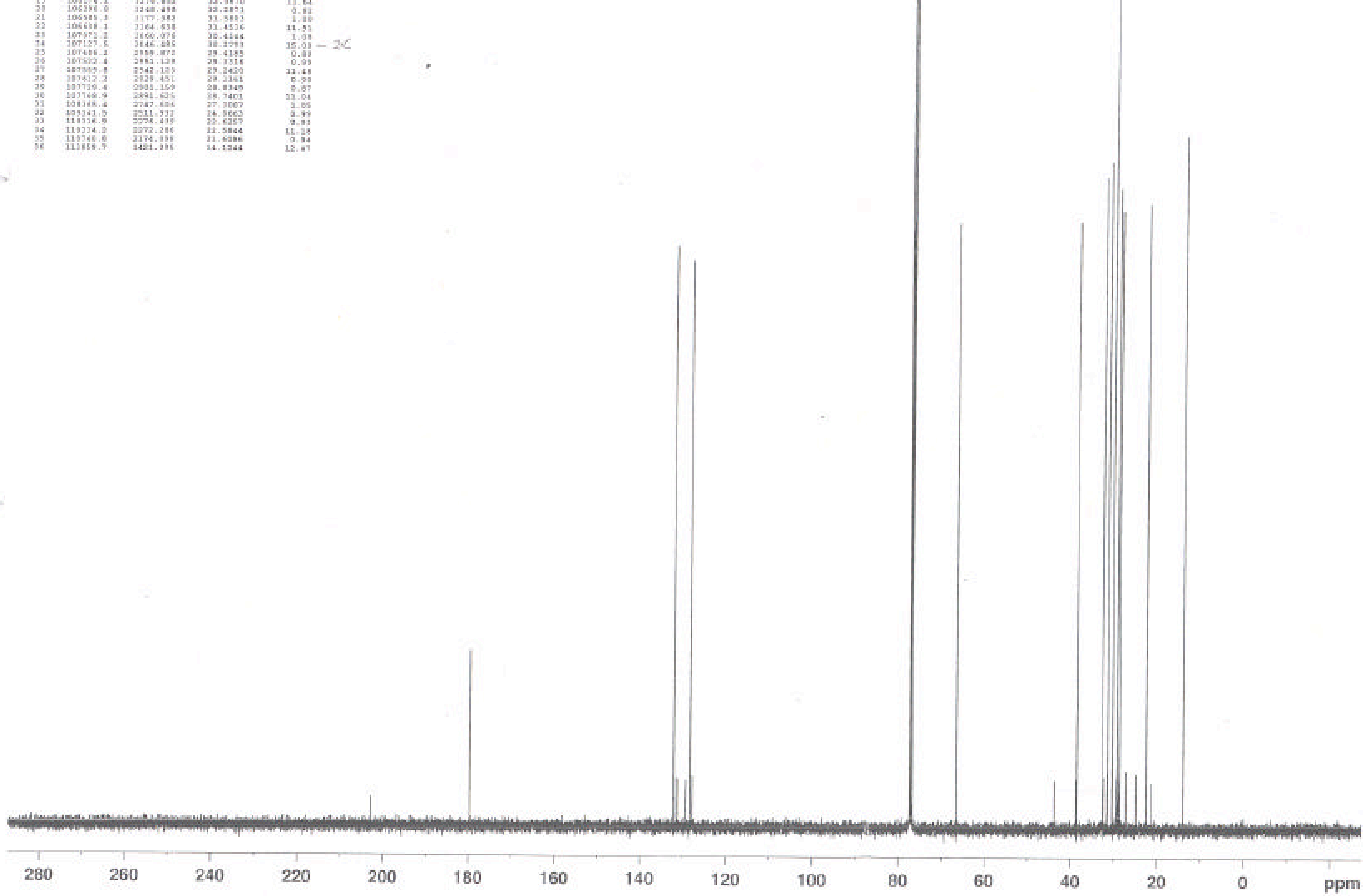
======== CHANNEL f1 ========
NUC1                       1H
P1                       9.90 usec
PL1                     -3.00 dB
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                     65536
SF             400.1300018 MHz
WDW                       GM
SSB                        0
LB                     -0.30 Hz
GB                        0.3
PC                       1.00
GR                       1.77 Hz
```

8-4064,Gruenanger,cg-277-8,CDCl3,TMS

pspin, 2008numerus, NAME=20080508m, EXPNO=64, PROCNO=1
ppm, F2=-27.179ppm, MI=0.20cm, MAXI=10000.00cm, PC=1.400

	ADDRESS	FREQUENCY [Hz]	[PPM]	INTENSITY
1	35219.4	20607.717	202.8345	0.53
2	44880.6	18075.166	179.6508	3.17
3	64654.2	13301.858	132.2009	18.37
4	64873.4	13248.436	131.6771	0.81
5	65082.0	13202.631	131.2222	0.87
6	65834.6	13021.338	129.4483	0.85
7	66237.7	12918.776	128.4018	10.36
8	66458.1	12867.343	127.8898	0.83
9	87482.8	7789.197	77.4176	16.99
10	87551.6	7777.661	77.3029	0.85
11	87616.1	7757.241	77.1008	17.43
12	87748.5	7725.280	76.7823	17.11
13	92028.8	6693.566	66.5123	10.90
14	92037.3	6689.325	66.4859	0.91
15	103478.5	4410.354	43.8349	0.89
16	103601.3	3897.845	38.7411	0.88
17	103671.3	3880.920	38.5729	10.86
18	106148.3	3300.836	32.8066	1.02
19	106174.2	3276.652	32.5670	11.64
20	106296.0	3248.488	32.2871	0.82
21	106585.3	3177.382	31.5803	1.00
22	106638.1	3164.638	31.4536	11.51
23	107072.2	3060.076	30.4144	1.09
24	107127.5	3046.886	30.2833	15.03 — 2C
25	107486.2	2959.872	29.4185	0.88
26	107522.4	2951.128	29.3316	0.99
27	107585.8	2942.135	29.2420	11.48
28	107612.2	2929.451	29.1161	0.98
29	107729.6	2901.159	28.8349	0.87
30	107768.9	2891.626	28.7401	11.06
31	108348.4	2747.404	27.3067	1.05
32	109341.5	2511.932	24.9663	0.99
33	110316.9	2276.435	22.6257	0.91
34	110334.2	2272.286	22.5844	11.18
35	110740.0	2174.098	21.6086	0.84
36	113859.7	1421.396	14.1344	12.47



BRUKER

Current Data Parameters
NAME 20080508m
EXPNO 64
PROCNO 1

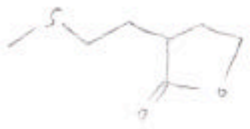
F2 - Acquisition Parameters
Date_ 20080508
Time 14.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1600
DS 2
SWH 31645.570 Hz
FIDRES 0.482873 Hz
AQ 1.0355188 sec
RG 16384
DW 15.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.90 usec
PL1 -3.00 dB
SFO1 100.6258487 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 131072
SF 100.6127605 MHz
WDW GM
SSB 0
LB -0.50 Hz
GB 0.1
PC 1.40
SR -8.53 Hz

GrBtM13-4010,Gruenanger,cg-282-3,CDC13,TM



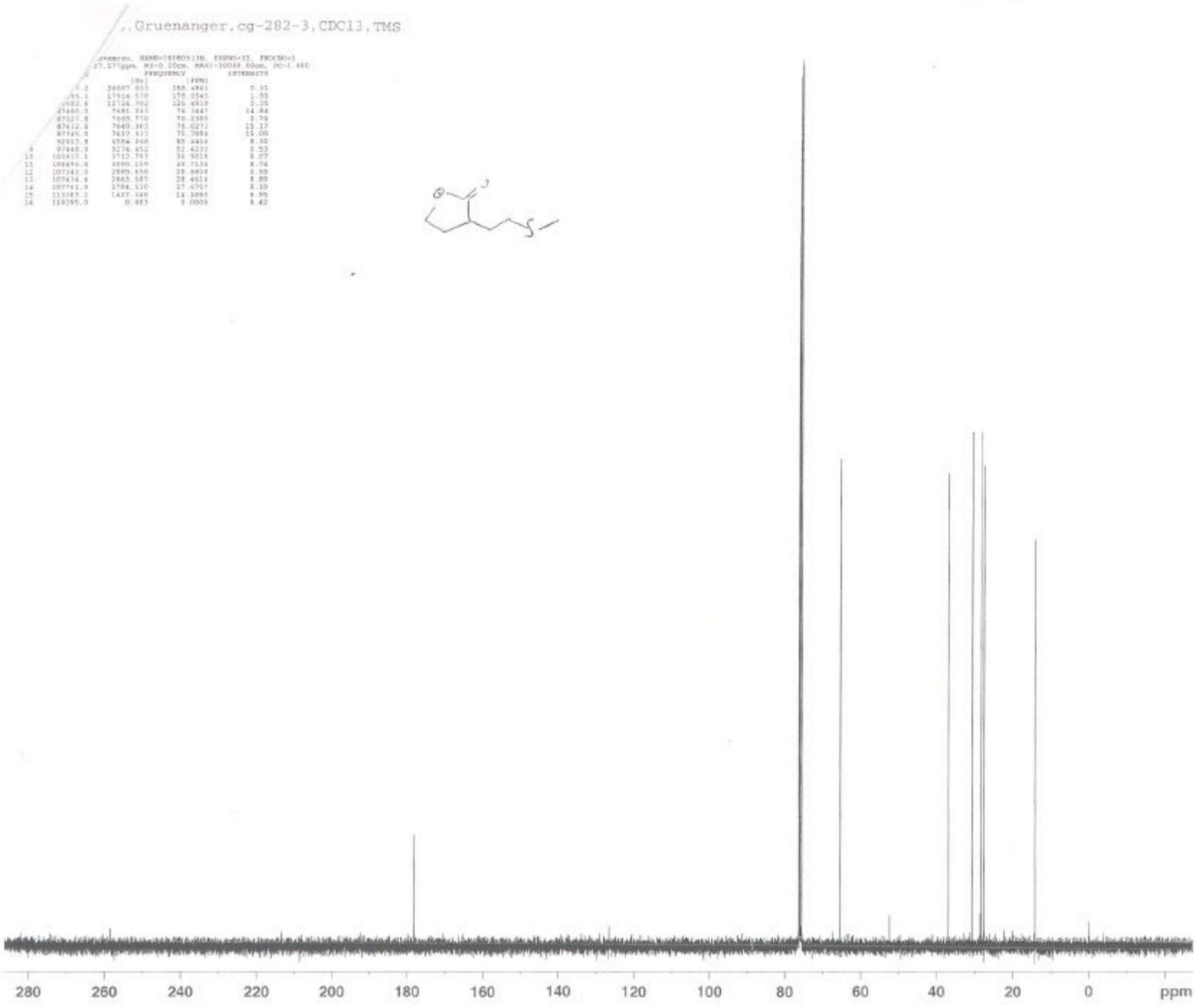
BRUKER

```
Current Data Parameters
NAME                  20080513B
EXPNO                        10
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20080513
Time                      7.40
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         64
DS                          2
SWH                  8278.146 Hz
FIDRES               0.126314 Hz
AQ                   3.9584243 sec
RG                       287.4
DW                      60.400 usec
DE                       6.00 usec
TE                      300.0 K
D1                 0.10000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                       9.90 usec
PL1                     -3.00 dB
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                      65536
SF             400.1300288 MHz
WDW                        GM
SSB                         0
LB                      -0.30 Hz
GB                         0.3
PC                        1.00
SR                       28.80 Hz
```



BRUKER

Current Data Parameters
NAME 20080513B
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080513
Time 8.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 31645.570 Hz
FIDRES 0.482873 Hz
AQ 1.0355188 sec
RG 1625.5
DW 15.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.90 usec
PL1 -3.00 dB
SFO1 100.6258487 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 131072
SF 100.6128693 MHz
WDW GM
SSB 0
LB -0.50 Hz
GB 0.1
PC 1.40
DR 100.26 Hz

GrBtM113-4020,Gruenanger,cg-281-4,CDCl3,TM

BRUKER

```
Current Data Parameters
NAME                  20080513B
EXPNO                       20
PROCNO                       1

F2 - Acquisition Parameters
Date_              20080513
Time                    8.55
INSTRUM                 spect
PROBHD     5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        64
DS                         2
SWH                 8278.146 Hz
FIDRES              0.126314 Hz
AQ                  3.9584243 sec
RG                      143.7
DW                     60.400 usec
DE                      6.00 usec
TE                     300.0 K
D1                 0.10000000 sec
TD0                         1

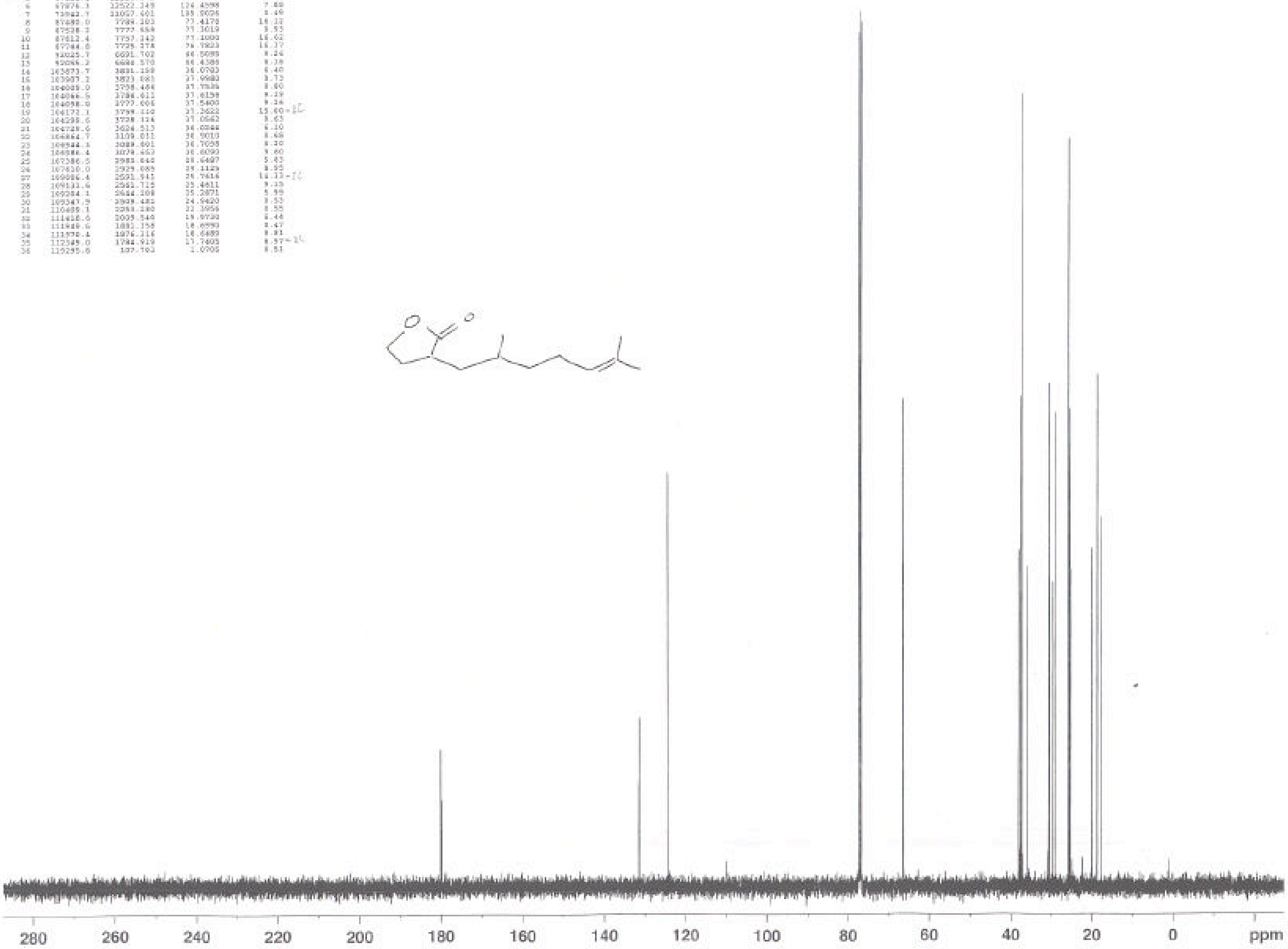
======== CHANNEL f1 ========
NUC1                       1H
P1                       9.90 usec
PL1                     -3.00 dB
SFO1             400.1324710 MHz

F2 - Processing parameters
SI                     65536
SF                400.1300291 MHz
WDW                       EM
SSB                        0
LB                     -0.30 Hz
GB                        0.3
PC                       1.00
SR                      29.14 Hz
```

-4022,Gruenanger,cg-281-4,CDCl3,TMS

dpin, USER=nmrsu, NAME=20080513B, EXPNO=22, PROCNO=1
ppm, F2=-17.388ppm, MI=0.28cm, MAXI=10000.00cm, PC=1.400

	ADDRESS	FREQUENCY [Hz]	[PPM]	INTENSITY
1	44693.4	18113.457	180.0510	2.63
2	44779.5	18098.472	179.8845	1.69
3	64906.0	13239.199	131.5677	2.62
4	64935.3	13232.128	131.5176	3.20
5	67056.0	12526.972	124.5068	5.12
6	67076.3	12522.149	124.4598	7.88
7	73942.7	11057.601	109.9026	2.49
8	87480.0	7789.203	77.4175	18.32
9	87528.3	7777.684	77.3019	9.53
10	87612.4	7757.242	77.1000	16.62
11	87784.8	7725.278	76.7823	16.37
12	92023.7	6691.702	66.5095	9.26
13	92056.2	6684.570	66.4385	9.38
14	103873.7	3831.159	38.0783	6.40
15	103907.2	3823.081	37.9980	3.73
16	104005.0	3798.484	37.7535	8.80
17	104066.5	3788.611	37.6159	9.29
18	104098.0	3777.006	37.5400	9.24
19	104172.1	3759.110	37.3622	15.60 ← 1C
20	104295.6	3728.124	37.0562	3.63
21	104725.6	3624.513	36.0266	6.30
22	106864.7	3109.011	30.9010	8.66
23	106944.3	3089.601	30.7058	8.20
24	106986.4	3079.453	30.6080	9.60
25	107386.5	2983.640	29.6487	5.43
26	107610.0	2929.085	29.1129	8.95
27	109086.4	2591.941	25.7616	11.13 ← 1C
28	109131.6	2561.715	25.4611	9.33
29	109284.1	2544.108	25.2871	5.99
30	109347.9	2509.481	24.9420	8.53
31	110488.1	2253.180	22.3956	8.55
32	111418.0	2009.549	19.9730	8.44
33	111889.6	1881.158	18.6993	8.47
34	111970.4	1876.116	18.6489	8.31
35	112349.0	1784.919	17.7405	8.97 ← 1C
36	115295.6	107.763	1.0705	8.51



BRUKER

Current Data Parameters
NAME 20080513B
EXPNO 22
PROCNO 1

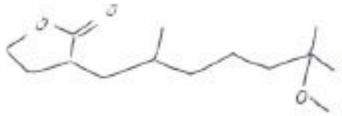
F2 - Acquisition Parameters
Date_ 20080513
Time 9.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 640
DS 2
SWH 31645.570 Hz
FIDRES 0.482873 Hz
AQ 1.0355188 sec
RG 7298.2
DW 15.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.90 usec
PL1 -3.00 dB
SFO1 100.6258487 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 131072
SF 100.6127614 MHz
WDW EM
SSB 0
LB -0.50 Hz
GB 0.1
PC 1.40
SR -7.63 Hz

GrBtMi13-4040,Gruenanger,cg-281-2,CDCl3,TM



BRUKER

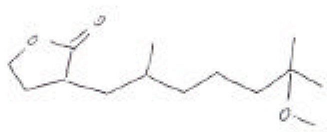
```
Current Data Parameters
NAME                  20080513B
EXPNO                        40
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20080513
Time                      10.51
INSTRUM                   spect
PROBHD     5 mm PABBO BB-
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          64
DS                           2
SWH                   8278.146 Hz
FIDRES                0.126314 Hz
AQ                    3.9584243 sec
RG                         128
DW                      60.400 usec
DE                       6.00 usec
TE                      300.0 K
D1                0.10000000 sec
TD0                          1

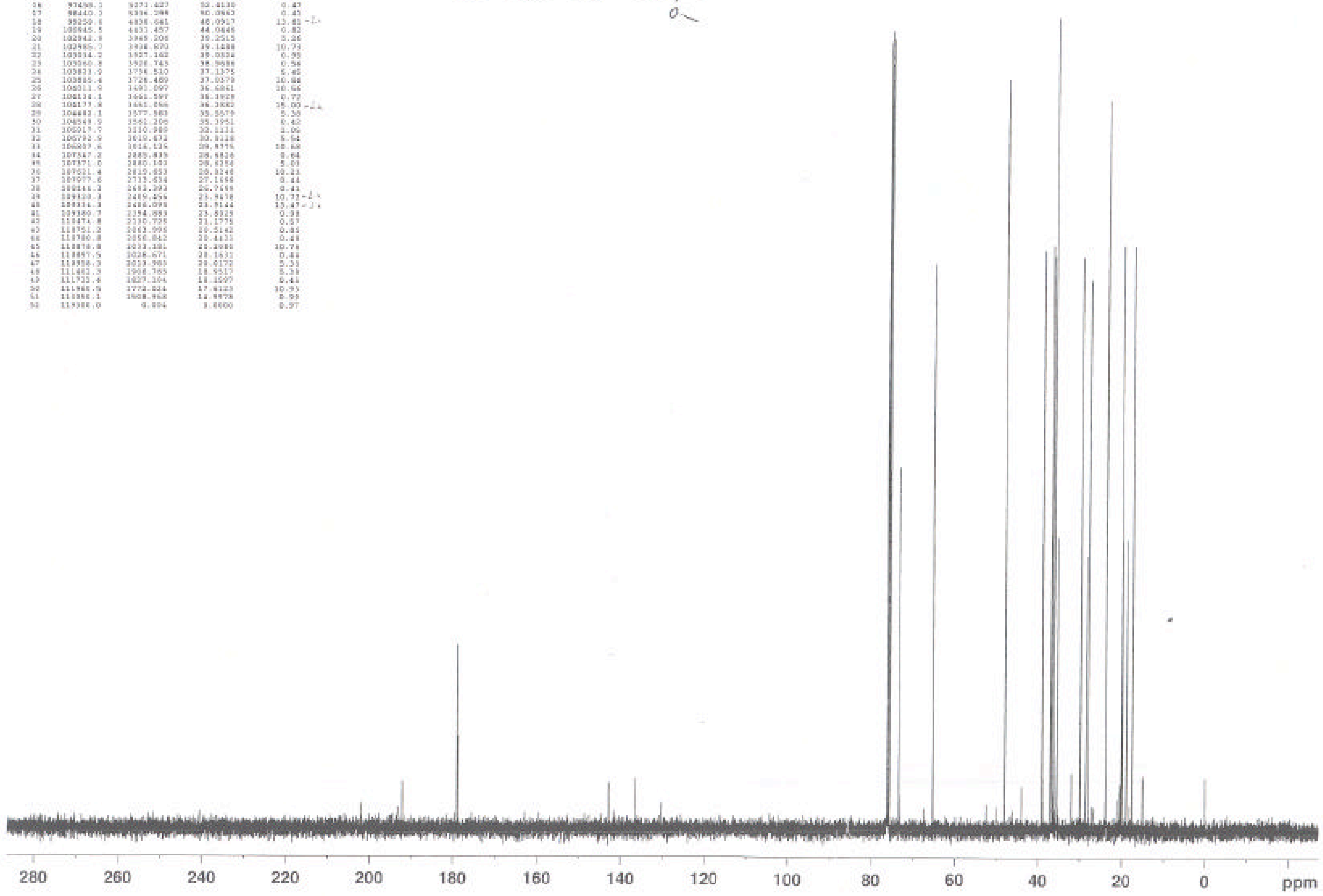
======== CHANNEL f1 ========
NUC1                        1H
P1                        9.90 usec
PL1                      -3.00 dB
SFO1             400.1324710 MHz

F2 - Processing parameters
SI                       65536
SF                400.1300160 MHz
WDW                         GM
SSB                          0
LB                       -0.30 Hz
GB                          0.3
PC                         1.00
SR                        25.99 Hz
```

.3-4042,Gruenanger,cg-281-2,CDC13,TMS



#	ADDRESS	FREQUENCY [Hz]	[ppm]	INTENSITY
1	35894.1	20338.170	202.0643	0.47
2	38220.2	19354.255	192.3442	0.87
3	44859.0	18011.416	179.0170	1.57
4	44784.8	17999.469	178.8110	1.71
5	59759.1	14375.259	142.8779	6.05
6	62457.1	13731.672	136.4993	6.90
7	65022.1	13109.304	130.2989	6.48
8	87482.1	7681.979	76.3519	14.93
9	87539.7	7670.273	76.2350	8.51
10	87614.6	7650.005	76.0341	14.78
11	87747.1	7618.025	75.7162	14.58
12	88858.7	7398.895	73.5383	6.65
13	88731.2	7380.435	73.3546	6.65
14	92028.7	6584.292	65.4418	10.47
15	92062.1	6576.237	65.3618	5.20
16	97498.1	5271.427	52.4130	0.47
17	98440.3	5036.298	50.0563	0.43
18	99250.6	4838.061	48.0917	13.81
19	100845.5	4433.457	44.0646	0.82
20	102942.8	3949.208	39.2513	5.26
21	102985.7	3938.870	39.1488	10.73
22	103034.2	3927.162	39.0324	0.55
23	103060.8	3920.745	38.9686	0.56
24	103821.9	3736.510	37.1375	5.45
25	103885.4	3728.489	37.0579	10.88
26	104011.9	3691.097	36.6861	10.66
27	104134.1	3661.597	36.3929	0.72
28	104177.8	3661.056	36.3882	15.00
29	104482.1	3577.583	35.5579	5.30
30	104548.9	3561.208	35.3951	0.42
31	105917.7	3230.989	32.1111	1.06
32	106792.9	3019.872	30.0118	5.54
33	106857.6	3016.135	29.9775	10.88
34	107547.2	2885.835	28.6826	0.64
35	107571.0	2880.103	28.6254	5.01
36	107621.4	2815.853	28.3246	10.21
37	107977.6	2733.634	27.1688	0.44
38	108146.3	2693.393	26.7699	0.41
39	109120.3	2409.256	23.9470	10.72
40	109334.3	2406.099	23.9144	15.47
41	109389.7	2354.883	23.8325	0.38
42	110474.8	2130.725	21.1775	0.57
43	110751.2	2063.995	20.5142	0.85
44	110780.8	2056.842	20.4433	0.48
45	110878.8	2033.181	20.2080	10.78
46	110897.5	2028.671	20.1632	0.84
47	110958.3	2013.985	20.0172	5.33
48	111482.3	1908.765	18.9517	5.38
49	111721.6	1827.104	18.1597	0.41
50	111960.5	1772.024	17.6123	10.95
51	111981.1	1508.968	14.9978	0.99
52	119388.0	0.806	0.0000	0.97



BRUKER

Current Data Parameters
NAME 20080513B
EXPNO 42
PROCNO 1

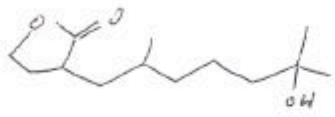
F2 - Acquisition Parameters
Date_ 20080513
Time 11.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 31645.570 Hz
FIDRES 0.482873 Hz
AQ 1.0355188 sec
RG 1149.4
DW 15.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.90 usec
PL1 -3.00 dB
SFO1 100.6258487 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 131072
SF 100.6128681 MHz
WDW GM
SSB 0
LB -0.50 Hz
GB 0.1
PC 1.40
SR 99.07 Hz

GrBtMi14-4030,Gruenanger,cg-283-8,CDCl3,TM



BRUKER

```
Current Data Parameters
NAME                  20080514B
EXPNO                        30
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20080514
Time                      10.20
INSTRUM                   spect
PROBHD     5 mm PABBO BB-
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          64
DS                           2
SWH                 8278.146 Hz
FIDRES              0.126314 Hz
AQ                  3.9584243 sec
RG                         256
DW                    60.400 usec
DE                     6.00 usec
TE                     300.0 K
D1                0.10000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       1H
P1                      9.90 usec
PL1                    -3.00 dB
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                       65536
SF             400.1300010 MHz
WDW                        EM
SSB                         0
LB                     -0.30 Hz
GB                        0.3
PC                       1.00
SR                       1.02 Hz
```

14-4031,Gruenanger,cg-283-8,CDCl3,TMS

[illegible]

	ADDRESS	FREQUENCY [Hz]	[PPM]	INTENSITY
1	44760.0	18118.609	180.0826	5.15
2	44776.3	18100.201	179.8957	3.14
3	87489.2	7788.201	77.4174	79.71
4	87511.4	7757.568	77.1020	3.11
5	87615.5	7757.363	77.1000	81.91
6	87747.9	7725.276	76.7822	78.85
7	90116.3	7148.645	71.0511	12.23 ~2C
8	92021.8	6693.475	66.5275	14.11
9	92052.2	6686.075	66.4538	9.50
10	101347.2	4441.908	44.1186	8.89
11	101380.3	4438.005	44.1108	13.77
12	103840.6	3839.918	38.1653	8.44
13	103916.6	3821.812	37.9854	14.65
14	104020.5	3796.497	37.7138	14.21
15	104261.4	3737.650	37.1475	14.04 ~2C
16	104508.4	3678.678	36.5627	8.51
17	106808.7	3183.306	31.6428	8.18
18	106838.1	3116.208	30.9723	13.34
19	107164.6	2989.101	29.7090	8.36
20	107483.6	2960.372	29.4236	13.16
21	107497.2	2957.088	29.3908	8.90
22	107515.8	2932.128	29.1514	8.81
23	107533.7	2928.266	29.1031	13.88
24	107610.6	2929.704	29.1106	13.26
25	110696.7	2184.615	21.7131	16.07
26	110777.4	2165.133	21.5196	9.63
27	111399.3	2016.873	20.0380	9.85
28	111957.7	1856.166	18.4471	15.56

BRUKER

Current Data Parameters
NAME 20080514B
EXPNO 31
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080514
Time 10.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1200
DS 2
SWH 31645.570 Hz
FIDRES 0.482873 Hz
AQ 1.0355188 sec
RG 1024
DW 15.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.90 usec
PL1 -3.00 dB
SFO1 100.6258487 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 131072
SF 100.6127606 MHz
WDW GM
SSB 0
LB -0.50 Hz
GB 0.1
PC 1.40
SR -8.39 Hz