

Highly Effective Vinylogous Mukaiyama-Michael Reaction Catalyzed by Silyl Methide Species Generated from 1,1,3,3-Tetrakis(trifluoromethanesulfonyl)propane

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1. General and materials

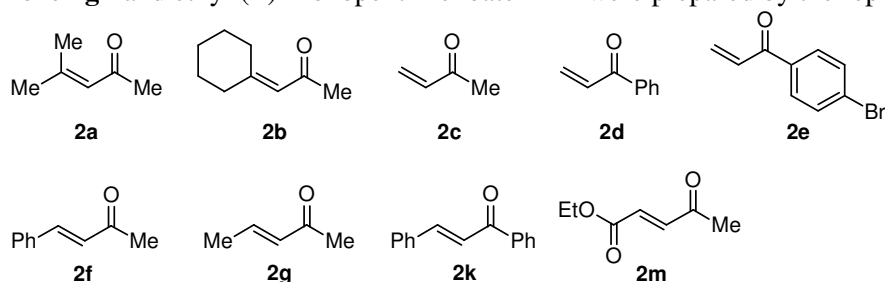
Melting points were uncorrected. ¹H and ¹³C NMR spectra were taken on a 400 MHz spectrometer, and chemical shifts were reported in parts per million (ppm) using CHCl₃ (7.26 ppm) in CDCl₃ or C₆D₅H (7.16 ppm) in C₆D₆ for ¹H NMR, and CDCl₃ (77.01 ppm) or C₆D₆ (128.00 ppm) for ¹³C NMR as an internal standard, respectively. Mass spectra were recorded by an electrospray ionization-time of flight (ESI-TOF) mass spectrometer. Column chromatography was performed on silica gel (75-150 μ m). Medium-pressure liquid chromatography (MPLC) was performed on a 30 x 4 cm i.d. prepacked column (silica gel, 50 μ m) with a UV detector.

2. Preparation of starting materials

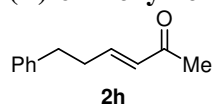
2.1. Preparation of α,β -enone substrates

4-Methylpent-3-en-2-one **2a**, but-3-en-2-one **2c**, (*E*)-4-phenylbut-3-en-2-one **2f** and (*E*)-chalcone **2k** are available commercially. 1-Cyclohexylideneacetone **2b**,¹ 1-phenylprop-2-en-1-one **2d**,² 1-(4-bromophenyl)-prop-2-en-1-one

2e,³ (*E*)-pent-3-en-2-one **2g**⁴ and ethyl (*E*)-4-oxopent-2-enoate **2m**⁵ were prepared by the reported procedure.

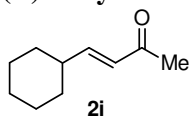


(*E*)-6-Phenylhex-3-en-2-one (**2h**)



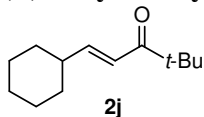
To a solution of 2-phenylpropanal (1.3 mL, 10 mmol) in toluene (50 mL), (2-oxopropyl)triphenylphosphonium chloride (4.0 g, 12 mmol) and K_2CO_3 (2.1 g, 15 mmol) were added at room temperature. After being stirred at 80 °C for 5 h, the aqueous layer was extracted with EtOAc (20 mL x 3). The combined organic layer was dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. Purification of the residue by column chromatography (hexane/EtOAc = 10 : 1) gave α,β -enone **2h** in 64% yield (1.1 g, 8.4 mmol) as colorless oil. The structure was confirmed by comparison to the reported spectrum data.⁶

(*E*)-4-Cyclohexylbut-3-en-2-one (**2i**)



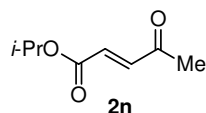
According to the synthetic procedure for **2h**, α,β -enone **2i** was prepared in 56% yield (1.3 g, 8.4 mmol) by the reaction of cyclohexanecarbaldehyde (1.8 mL, 15 mmol), (2-oxopropyl)triphenylphosphonium chloride (8.0 g, 23 mmol) and K_2CO_3 (4.2 g, 30 mmol) in a mixed solvent of CH_2Cl_2 (25 mL) and H_2O (25 mL) at 50 °C for 24 h. The structure was confirmed by comparison to the reported spectrum data.⁷

(*E*)-1-Cyclohexyl-4,4-dimethylpent-1-en-3-one (**2j**)



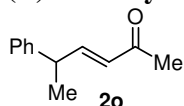
To a solution of cyclohexanecarbaldehyde (2.5 mL, 20 mmol) in a mixed solvent of EtOH (8 mL) and H_2O (3 mL), 3,3-dimethylbutan-2-one (7.3 mL, 24 mmol) and 0.25 M aqueous solution of NaOH (2 mL, 5 mmol) were added at room temperature. After being stirred at the same temperature for 50 h, the reaction mixture was diluted with H_2O (30 mL) and extracted with EtOAc (50 mL x 3). The combined organic layer was washed with brine (50 mL), dried over anhydrous $MgSO_4$ and evaporated. The obtained residue was purified by column chromatography on silica gel (hexane/EtOAc = 20 : 1) to give α,β -enone **2j** in 46% yield (1.8 g, 9.2 mmol) as colorless oil. The structure was confirmed by comparison to the reported data.⁸

Isopropyl (*E*)-4-oxopent-2-enoate (**2n**)



A solution of ethyl 4-oxopentanoate (5.7 mL, 40 mmol) in propan-2-ol (25 mL) was treated by a catalytic amount of iodine (250 mg, 0.97 mmol) for 7 days at 100 °C, then the reaction mixture was evaporated to give a crude mixture of isopropyl 4-oxopentanoate. To a solution of this crude mixture in Et₂O (50 mL), bromine (1.2 mL, 23 mmol) was added at 0 °C. After being stirred at the same temperature for 3 h, the resultant mixture was quenched with H₂O (30 mL) and extracted with Et₂O (20 mL x 3). The organic layer was dried over anhydrous MgSO₄ and concentrated under reduced pressure to give a crude mixture of isopropyl 3-bromo-4-oxopentanoate. A crude mixture was dissolved in AcOH (1.9 mL) and treated with NaOAc (2.7 g) at 80 °C for 2 h. After usual extractive work up, the reaction mixture was purified by column chromatography on silica gel (pentane/Et₂O = 10 : 1) to give **2n** (874 mg, 5.6 mmol, 14 % yield over 3 steps). Colorless oil; IR (neat) ν 2984, 1722, 1702, 1687, 1108, 980 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 1.29 (6H, d, *J* = 6.2 Hz), 2.35 (3H, s), 5.12 (1H, septet, *J* = 6.2 Hz), 6.62 (1H, d, *J* = 16.1 Hz), 6.99 (1H, d, *J* = 16.1 Hz); ¹³C NMR (100 MHz, CDCl₃) δ 21.4, 27.6, 68.7, 131.8, 139.5, 164.6, 197.3; MS (ESI-TOF) *m/z* 157 [M+H]⁺; HRMS calcd for C₈H₁₃O₃ [M+H]⁺, 157.0865; found, 157.0859;

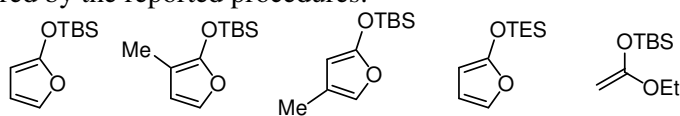
(*E*)-5-Phenylhex-3-en-2-one (**2o**)



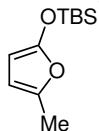
According to the synthetic procedure for **2h**, α,β -enone **2o** was prepared in 64% yield (1.7 g, 6.4 mmol) by the reaction of 2-phenylpropanal (1.3 mL, 10 mmol), (2-oxopropyl)triphenylphosphonium chloride (4.0 g, 12 mmol), and K₂CO₃ (2.1 g, 15 mmol) in toluene (50 mL) at 80 °C for 5 h. The structure was confirmed by comparison to the reported spectrum data.⁹

2.2. Preparation of 2-silyloxyfurans and ketene silyl acetal

tert-Butyl(furan-2-yloxy)dimethylsilane,¹⁰ *tert*-butyl(dimethyl)[(3-methylfuran-2-yl)oxy]silane,¹¹ and *tert*-butyl(dimethyl)[(4-methylfuran-2-yl)oxy]silane,¹¹ triethyl(furan-2-yloxy)silane,¹² and *tert*-butyl(1-ethoxyvinlyloxy)-dimethylsilane¹³ were prepared by the reported procedures.



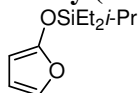
tert-Butyl(dimethyl)[(5-methylfuran-2-yl)oxy]silane



To a solution of 5-methylfuran-2(3*H*)-one (0.9 mL, 10 mmol) in CH₂Cl₂ (2 mL), *tert*-butyl(dimethyl)silyl trifluoromethanesulfonate (2.1 mL, 10 mmol) and Et₃N (2.1 mL 15 mmol) were added at 0 °C. After being stirred at room temperature for 2 h, extractive work-up and purification by silica gel column chromatography (hexane) gave *tert*-butyl(dimethyl)[(5-methylfuran-2-yl)oxy]silane (1.27 g, 6.0 mmol, 60% yield). Colorless oil; IR

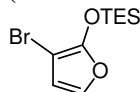
(neat) ν 1628, 1592 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 0.27 (6H, s), 1.01 (9H, s), 2.19 (3H, s), 4.99 (1H, d, J = 3.0 Hz), 5.75-5.80 (1H, m); ^{13}C NMR (100.6 MHz, CDCl_3) δ -4.9, 13.5, 18.1, 25.6, 83.7, 106.1, 141.3, 155.3; MS (EI) m/z 212 $[\text{M}]^+$. Anal. Calcd for $\text{C}_{11}\text{H}_{20}\text{O}_2\text{Si}$: C, 62.21; H, 9.49. Found: C, 61.97; H, 9.25.

Diethyl(furan-2-yloxy)isopropylsilane



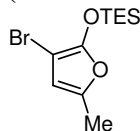
Furan-2(5*H*)-one (0.7 mL, 10 mmol) and Et_3N (2.1 mL 15 mmol) were added diethylisopropylsilyl chloride (1.8 mL, 10 mmol) at room temperature. After being stirred at the same temperature for 2 h, the desired silyloxyfuran was obtained in 71 % yield (1.6 g, 7.1 mmol) by direct distillation of the reaction mixture under reduced pressure (bp. 80 °C at 3 mmHg). Colorless oil; IR (neat) ν 2958, 2879, 1619, 1523, 1261, 957, 837, 735, 708 cm^{-1} ; ^1H NMR (400 MHz, C_6D_6) δ 0.60-0.77 (4H, m), 0.92-1.03 (13H, m), 5.11 (1H, d, J = 3.0 Hz), 6.02-6.08 (1H, m), 6.62-6.68 (1H, m); ^{13}C NMR (100 MHz, C_6D_6) δ 4.4, 7.3, 13.7, 17.5, 84.4, 112.0, 133.0, 158.1; MS (EI) m/z 212 $[\text{M}]^+$.

(3-Bromofuran-2-yloxy)triethylsilane



To a solution of 3-bromofuran-2(5*H*)-one¹⁴ (490 mg, 3.0 mmol) in CH_2Cl_2 (0.5 mL), triethylsilyl triflate (0.68 mL, 3.0 mmol) and Et_3N (0.46 mL 3.6 mmol) were added at 0 °C. After being stirred at 0 °C for 15 min, the reaction mixture was diluted with hexane (20 mL), then washed with H_2O (10 mL). The resultant organic layer was evaporated to give (3-bromofuran-2-yloxy)triethylsilane in 82% yield (680 mg, 2.5 mmol) as a dark red oil. Due to the low stability of this compound, the crude mixture was used without further purifications (>94% purity based on ^1H NMR). IR (neat) ν 2958, 2914, 2879, 1636, 1518, 886, 839, 749 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 0.74-0.82 (6H, m), 0.98-1.02 (9H, m), 6.26 (1H, d, J = 2.4 Hz), 6.80 (1H, d, J = 2.4 Hz); ^{13}C NMR (100 MHz, CDCl_3) δ 4.9, 6.2, 73.6, 114.1, 132.1, 153.4; MS (EI) m/z 276 $[\text{M}]^+$.

(3-Bromo-5-methylfuran-2-yloxy)triethylsilane



To a solution of 5-methylfuran-2(5*H*)-one¹⁵ (1.0 g, 10 mmol) in CCl_4 (40 mL), bromine (0.8 mL, 15 mmol) was added at room temperature. After being stirred at 50 °C for 2 h, the resultant mixture was quenched with saturated NaHCO_3 aqueous solution (30 mL) and extracted with CH_2Cl_2 (20 mL x 3). The organic layer was dried over anhydrous MgSO_4 and concentrated under reduced pressure to give a crude mixture of 3,4-dibromo-5-methyldihydrofuran-2(3*H*)-one. This crude mixture was dissolved to CH_2Cl_2 (40 mL) and treated with Et_3N (1.1 mL) at 0 °C for 30 min. After usual extractive work up, the reaction mixture was purified by column chromatography on silica gel (hexane/ AcOEt = 3 : 1) to give 3-bromo-5-methylfuran-2(5*H*)-one (1.4 g, 8.2 mmol, 82 % yield over 2 steps). Pale yellow oil; IR (neat) ν 3092, 2985, 2935, 1767, 1608, 1314, 993 cm^{-1} ;

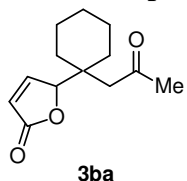
^1H NMR (400 MHz, CDCl_3) δ 1.49 (3H, d, J = 6.8 Hz), 5.08 (1H, qd, J = 6.8, 1.6 Hz), 7.50 (1H, d, J = 1.6 Hz); ^{13}C NMR (100 MHz, CDCl_3) δ 18.5, 79.2, 112.6, 154.1, 168.2; MS (ESI-TOF) m/z 177 $[\text{M}+\text{H}]^+$, 179 $[\text{M}+2+\text{H}]^+$; HRMS calcd for $\text{C}_5\text{H}_6\text{BrO}_2$ $[\text{M}+\text{H}]^+$, 176.9551; found, 176.9544; Anal. Calcd for $\text{C}_5\text{H}_5\text{BrO}_2$: C, 33.93; H, 2.85. Found: C, 34.05; H, 3.01.

According to the synthetic procedure for (3-bromofuran-2-yloxy)triethylsilane, (3-bromo-5-methylfuran-2-yloxy)triethylsilane was obtained in 90% yield (780 mg, 2.7 mmol, >90% purity based on ^1H NMR) by the reaction of 3-bromo-5-methylfuran-2(5*H*)-one (530 mg, 3.0 mmol), triethylsilyl triflate (0.68 mL, 3.0 mmol) and Et_3N (0.46 mL, 3.6 mmol) in CH_2Cl_2 (0.5 mL). Colorless oil; IR (neat) ν 2957, 2916, 2879, 1641, 1598, 1057, 854 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 0.78 (6H, q, J = 7.9 Hz), 1.02 (9H, t, J = 7.9 Hz), 2.15 (1H, brs), 5.83 (1H, brs); ^{13}C NMR (100 MHz, CDCl_3) δ 4.9, 6.2, 13.6, 73.3, 109.1, 141.3, 151.7. Due to low stability of this compound, it was not possible to measure its MS spectrum.

3. Vinylogous Mukaiyama-Michael (VMM) reaction

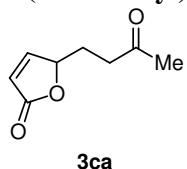
3.1. Carbon acid catalyzed VMM reaction (Tables 1, 2)

5-[1-(2-Oxopropyl)cyclohexyl]furan-2(5*H*)-one (3ba)



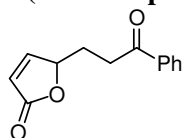
According to the synthesis of **3aa**, **3ba** was obtained in 90% yield (100.0 mg, 0.45 mmol) by the reaction of (*tert*-butyldimethylsilyloxy)furan (109 mg, 0.55 mmol) and **2b** (69 mg, 0.50 mmol) in the presence of **1** (0.74 mg, 1.3 μmol) at -24°C for 3 h. Colorless oil; IR (neat) ν 1757, 1713 cm^{-1} ; ^1H NMR (400MHz, CDCl_3) δ 1.10-1.45 (5H, m), 1.45-1.63 (4H, m), 1.74-1.82 (1H, m), 2.12 (3H, s), 2.54 (1H, d, J = 17.4 Hz), 2.67 (1H, d, J = 17.4 Hz), 5.34-5.37 (1H, m), 6.07 (1H, dd, J = 5.8, 2.1 Hz), 7.51 (1H, dd, J = 5.8, 1.4 Hz); ^{13}C NMR (100.6 MHz, CDCl_3) δ 21.0, 21.1, 25.6, 30.5, 30.6, 32.1, 40.3, 43.6, 87.7, 121.9, 154.7, 172.9, 208.2; MS (ESI-TOF) m/z 223 $[\text{M}+\text{H}]^+$; HRMS calcd for $\text{C}_{13}\text{H}_{19}\text{O}_3$ $[\text{M}+\text{H}]^+$, 223.1334; found, 223.1320. Anal. Calcd for $\text{C}_{13}\text{H}_{18}\text{O}_3$: C, 70.24; H, 8.16. Found: C, 70.45; H, 8.12.

5-(3-Oxobutyl)furan-2(5*H*)-one (3ca)



According to the synthesis of **3aa**, the reaction of **2c** (35 mg, 0.50 mmol) and *tert*-butyl(furan-2-yloxy)dimethylsilane (109 mg, 0.55 mmol) in the presence of **1** (0.14 mg, 0.25 μmol) for 2 h at -78°C give 1,4-adduct **3ca** (63.1 mg, 0.41 mmol, 82% yield). The structure was confirmed by comparison of spectrum data with those reported in the literature.¹⁶

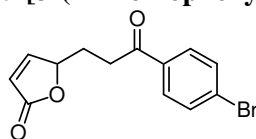
5-(3-Oxo-3-phenylpropyl)furan-2(5H)-one (3da)



3da

To a solution of *tert*-butyl(furan-2-yloxy)dimethylsilane (109 mg, 0.55 mmol) in CH₂Cl₂ (1 mL), a solution of **1** (1.5 mM in CH₂Cl₂, 2.0 mL, 0.50 μmol) and **2d** (66 mg, 0.50 mmol) in CH₂Cl₂ (0.8 mL) were added at –78 °C over 10 min. After being stirred for 1 h at –78 °C, reaction mixture was quenched with saturated NaHCO₃ aqueous solution and extracted with EtOAc (20 mL x 3). The organic layer was dried over anhydrous MgSO₄, then it was concentrated under reduced pressure. The resulting residue was treated by TfOH (150 mg, 1.0 mmol) in CH₂Cl₂ (100 mL) for 1 h at –78 °C. The reaction mixture was diluted with H₂O (50 mL), extracted with CH₂Cl₂ (40 mL), dried over anhydrous MgSO₄ and evaporated. Purification of the resulting residue by column chromatography on silica gel (hexane/EtOAc = 3 : 1) gave **3da** (96.2 mg, 0.45 mmol, 89% yield). White solid; Mp. 91.1-91.5 °C; IR (KBr) ν 1769, 1682 cm⁻¹; ¹H NMR (400MHz, CDCl₃) δ 1.97-2.03 (1H, m), 2.37-2.48 (1H, m), 3.08-3.28 (2H, m), 5.17-5.27 (1H, m), 6.11 (1H, dd, *J* = 5.7, 2.0 Hz), 7.44-7.52 (3H, m), 7.55-7.61 (1H, m), 7.93-7.98 (2H, m); ¹³C NMR (100.6 MHz, CDCl₃) δ 27.1, 33.2, 82.3, 121.6, 127.9, 128.6, 133.3, 136.4, 156.2, 172.8, 198.5; MS (ESI-TOF) *m/z* 239 [M+Na]⁺; HRMS calcd for C₁₃H₁₂NaO₃ [M+Na]⁺, 239.0684; found, 239.0671.

5-[3-(4-Bromophenyl)-3-oxopropyl]furan-2(5H)-one (3ea)

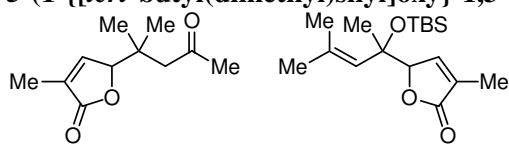


3ea

According to the synthetic procedure for **3da**, **3ea** was obtained in 88% yield (129.9 mg, 0.44 mmol) by the reaction of **2e** (106 mg, 0.50 mmol) and *tert*-butyl(furan-2-yloxy)dimethylsilane (109 mg, 0.55 mmol) in the presence of **1** (0.14 mg, 0.25 μmol) for 1 h at –78 °C. White solid; Mp. 84.3-84.9 °C; IR (KBr) ν 1746, 1687 cm⁻¹; ¹H NMR (400MHz, CDCl₃) δ 1.88-1.99 (1H, m), 2.40 (1H, dtd, *J* = 14.5, 7.3, 3.8 Hz), 3.03-3.23 (2H, m), 5.16-5.22 (1H, m), 6.12 (1H, dd, *J* = 5.7, 2.0 Hz), 7.49 (1H, dd, *J* = 5.7, 1.5 Hz), 7.58-7.63 (2H, m), 7.78-7.83 (2H, m); ¹³C NMR (100.6 MHz, CDCl₃) δ 27.1, 33.3, 82.1, 121.8, 128.7, 129.5, 132.0, 135.2, 156.0, 172.7, 197.5; MS (ESI-TOF) *m/z* 295 [M+H]⁺; HRMS calcd for C₁₃H₁₂BrO₃ [M+H]⁺, 294.9970; found, 294.9966. Anal. Calcd for C₁₃H₁₁BrO₃: C, 52.91; H, 3.76. Found: C, 52.61; H, 4.00.

5-(1,1-Dimethyl-3-oxobutyl)-3-methylfuran-2(5H)-one (3ab) and

5-(1-[[*tert*-butyl(dimethyl)silyl]oxy]-1,3-dimethylbut-2-enyl)-3-methylfuran-2(5H)-one (4ab).



3ab

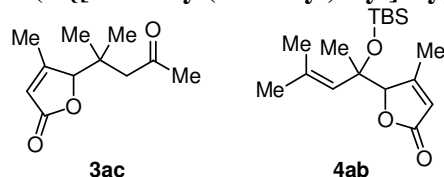
4ab

According to the synthetic procedure for **3aa**, the reaction of **2a** (49 mg, 0.50 mmol) and *tert*-butyl(dimethyl)[(3-methylfuran-2-yl)oxy]silane (117 mg, 0.55 mmol) in the presence of **1** (0.71 mg, 1.3 μmol) for 2 h at –78 °C

give 1,4-adduct **3ab** (82.3 mg, 0.42 mmol, 84% yield) and two diastereomers of 1,2-adduct **4ab** (major isomer, 2.1 mg, 0.007 mmol 1.3% yield; minor isomer, 2.0 mg 0.006 mmol, 1.3% yield). For **3ab** Colorless oil; IR (neat) ν 1756, 1713 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 0.89 (3H, s), 1.14 (3H, s), 1.92-1.94 (3H, m), 2.14 (3H, s), 2.38 (1H, d, $J = 16.9$ Hz), 2.65 (1H, d, $J = 16.9$ Hz), 5.03-5.08 (1H, m), 7.04 (1H, t, $J = 1.5$ Hz); ^{13}C NMR (100.6 MHz, CDCl_3) δ 10.8, 22.4, 23.3, 32.0, 37.1, 50.5, 86.1, 131.2, 146.4, 174.2, 207.7; MS (ESI-TOF) m/z 197 $[\text{M}+\text{H}]^+$; HRMS calcd for $\text{C}_{11}\text{H}_{17}\text{O}_3$ $[\text{M}+\text{H}]^+$, 197.1178; found, 197.1186. Anal. Calcd for $\text{C}_{11}\text{H}_{16}\text{O}_3$: C, 67.32; H, 8.22. Found: C, 67.30; H, 8.24. For **4ab-major** Colorless oil; IR (neat) ν 1767 cm^{-1} ; ^1H NMR (400 MHz, C_6D_6) δ 0.11 (6H, s), 0.96 (9H, s), 1.26 (3H, s), 1.52 (3H, d, $J = 1.3$ Hz), 1.64 (3H, t, $J = 1.8$ Hz), 1.69 (3H, d, $J = 1.3$ Hz), 4.47-4.51 (1H, m), 4.92-4.96 (1H, m), 6.42-6.46 (1H, m); ^{13}C NMR (100.6 MHz, C_6D_6) δ -2.3, -1.9, 10.6, 18.5, 19.4, 26.1, 26.1, 27.6, 77.2, 86.8, 127.6, 131.4, 136.4, 145.6, 173.2; MS (ESI-TOF) m/z 333 $[\text{M}+\text{Na}]^+$; HRMS calcd for $\text{C}_{17}\text{H}_{30}\text{NaO}_3\text{Si}$ $[\text{M}+\text{Na}]^+$, 333.1862; found, 333.1877. For **4ab-minor** Colorless oil; IR (neat) ν 1767 cm^{-1} ; ^1H NMR (400 MHz, C_6D_6) δ 0.06 (3H, s), 0.09 (3H, s), 0.91 (9H, s), 1.26 (3H, s), 1.55 (3H, d, $J = 1.3$ Hz), 1.58 (3H, d, $J = 1.3$ Hz), 1.69 (3H, t, $J = 1.7$ Hz), 4.46-4.50 (1H, m), 5.09-5.14 (1H, m), 6.48-6.52 (1H, m); ^{13}C NMR (100.6 MHz, C_6D_6) δ -2.3, -1.9, 10.7, 18.4, 19.5, 25.5, 26.1, 27.5, 76.3, 85.9, 129.7, 131.5, 135.4, 145.8, 173.4; MS (ESI-TOF) m/z 333 $[\text{M}+\text{Na}]^+$; HRMS calcd for $\text{C}_{17}\text{H}_{30}\text{NaO}_3\text{Si}$ $[\text{M}+\text{Na}]^+$, 333.1862; found, 333.1880.

5-(1,1-Dimethyl-3-oxobutyl)-4-methylfuran-2(5H)-one (**3ac**) and

5-(1-([*tert*-butyl(dimethyl)silyl]oxy)-1,3-dimethylbut-2-enyl)-4-methylfuran-2(5H)-one (**4ac**)

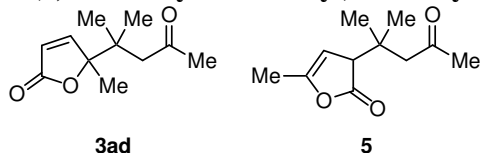


According to the synthetic procedure for **3aa**, the reaction of **2a** (49 mg, 0.50 mmol) and *tert*-butyl [(4-methylfuran-2-yl)oxy]silane (159 mg, 0.75 mmol) in the presence of **1** (0.71 mg, 1.3 μmol) for 2 h at -78 $^{\circ}\text{C}$ give 1,4-adduct **3ac** (72.0 mg, 0.39 mmol, 78% yield) and two diastereomers of 1,2-adduct **4ac** (major isomer, 9.4 mg, 0.03 mmol 6.3% yield; minor isomer, 2.6 mg, 0.009 mmol, 1.8% yield). For **3ac** Colorless oil; IR (neat) ν 1763, 1713 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 0.83 (3H, s), 1.18 (3H, s), 2.09 (3H, s), 2.13-2.15 (3H, m), 2.64 (1H, d, $J = 17.4$ Hz), 2.74 (1H, d, $J = 17.4$ Hz), 5.15 (1H, brs), 5.78 (1H, quin, $J = 1.5$ Hz); ^{13}C NMR (100.6 MHz, CDCl_3) δ 16.5, 22.4, 23.6, 31.5, 36.9, 51.7, 88.8, 119.1, 167.6, 172.6, 207.5; MS (ESI-TOF) m/z 219 $[\text{M}+\text{Na}]^+$; HRMS calcd for $\text{C}_{11}\text{H}_{16}\text{NaO}_3$ $[\text{M}+\text{Na}]^+$, 219.0997; found, 219.0977. Anal. Calcd for $\text{C}_{11}\text{H}_{16}\text{O}_3$: C, 67.32; H, 8.22. Found: C, 67.31; H, 8.21. For **4ac-major** Colorless oil; IR (neat) ν 1767 cm^{-1} ; ^1H NMR (400 MHz, C_6D_6) δ 0.03 (3H, s), 0.06 (3H, s), 0.89 (9H, s), 1.21 (3H, s), 1.52 (3H, s), 1.53 (3H, s), 1.67-1.70 (3H, m), 5.17 (1H, s), 5.28 (1H, s), 5.51 (1H, m); ^{13}C NMR (100.6 MHz, C_6D_6) δ -2.3, -1.8, 16.0, 18.4, 19.4, 24.9, 26.2, 27.4, 76.5, 88.4, 119.5, 131.1, 134.4, 166.5, 171.9; MS (ESI-TOF) m/z 333 $[\text{M}+\text{Na}]^+$; HRMS calcd for $\text{C}_{17}\text{H}_{30}\text{NaO}_3\text{Si}$ $[\text{M}+\text{Na}]^+$, 333.1862; found, 333.1881. For **4ac-minor** Colorless oil; IR (neat) ν 1769 cm^{-1} ; ^1H NMR (400 MHz, C_6D_6) δ 0.09 (3H, s), 0.11 (3H, s), 0.93 (9H, s), 1.40 (3H, s), 1.50 (3H, d, $J = 1.4$), 1.52-1.56 (3H, m), 1.70 (3H, d, $J = 1.4$ Hz), 4.31 (1H, brs), 4.88 (1H, brs), 5.43-5.46 (1H, m); ^{13}C NMR (100.6 MHz, C_6D_6) δ -2.1, -2.0, 16.1, 18.4, 19.2, 26.2, 26.7, 27.8, 77.9, 89.5, 119.3, 126.9, 136.7, 166.2, 171.5; MS

(ESI-TOF) m/z 333 $[M+Na]^+$; HRMS calcd for $C_{17}H_{30}NaO_3Si$ $[M+Na]^+$, 333.1862; found, 333.1854.

5-(1,1-Dimethyl-3-oxobutyl)-5-methylfuran-2(5H)-one (3ad) and

3-(1,1-dimethyl-3-oxobutyl)-5-methylfuran-2(3H)-one (5)

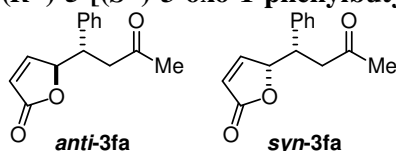


According to the synthetic procedure for **3aa**, the reaction of **2a** (49 mg, 0.50 mmol) and *tert*-butyl(dimethyl) [(5-methylfuran-2-yl)oxy]silane (159 mg, 0.75 mmol) in the presence of **1** (0.71 mg, 1.3 μ mol) for 2 h at -78 $^{\circ}$ C, then 2 h at -24 $^{\circ}$ C gave **3ad** (51.0 mg, 0.28 mmol, 55% yield) and **5** (27.0 mg, 0.15 mmol, 29% yield). For **3ad** White solid; Mp. 55.3 – 55.7 $^{\circ}$ C; IR (KBr) 1752 , 1705 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$) δ 1.04 (3H, s), 1.13 (3H, s), 1.39 (3H, s), 2.09 (3H, s), 2.40 (1H, d, $J = 15.5$ Hz), 2.46 (1H, d, $J = 15.5$ Hz), 6.00 (1H, d, $J = 5.8$ Hz), 7.46 (1H, d, $J = 5.8$ Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 19.8, 22.0, 22.5, 32.7, 39.6, 48.5, 93.5, 121.0, 159.5, 172.5, 207.7; MS (ESI-TOF) m/z 197 $[M+H]^+$; HRMS calcd for $C_{11}H_{17}O_3$ $[M+H]^+$, 197.1178; found, 197.1177. Anal. Calcd for $C_{11}H_{16}O_3$: C, 67.32; H, 8.22. Found: C, 67.33; H, 8.17. For **5** Colorless oil; IR (neat) ν 1789, 1713 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$) δ 0.96 (3H, s), 1.12 (3H, s), 1.97–2.02 (3H, m), 2.15 (3H, s), 2.44 (1H, d, $J = 17.2$ Hz), 2.91 (1H, d, $J = 17.2$ Hz), 3.68 (1H, quin, $J = 2.4$ Hz), 5.08–5.12 (1H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 14.0, 24.5, 25.6, 31.8, 35.7, 50.9, 52.2, 101.6, 152.5, 177.8, 208.0; MS (ESI-TOF) m/z 197 $[M+H]^+$; HRMS calcd for $C_{11}H_{17}O_3$ $[M+H]^+$, 197.1178; found, 197.1183. Anal. Calcd for $C_{11}H_{16}O_3$: C, 67.32; H, 8.22. Found: C, 67.52; H, 8.32.

3.2. Diastereoselective VMM reaction (Tables 3, 4 and Eqn. 1, 2)

(*S**)-5-[(*S**)-3-Oxo-1-phenylbutyl]furan-2(5H)-one (*anti*-3fa) and

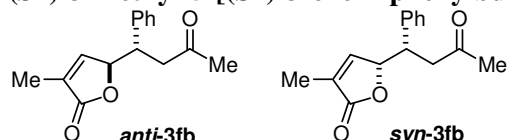
(*R**)-5-[(*S**)-3-oxo-1-phenylbutyl]furan-2(5H)-one (*syn*-3fa)



To a solution of α,β -enone **2f** (73 mg, 0.5 mmol) and triethyl(furan-2-yloxy)silane (119 mg, 0.60 mmol) in CH_2Cl_2 (0.5 mL), freshly prepared solution of carbon acid **1** in CH_2Cl_2 (10 mM, 0.5 mL, 5.0 μ mol) were added at -78 $^{\circ}$ C over 30 min. After being stirred for 2 h at -24 $^{\circ}$ C, the reaction mixture was quenched with saturated $NaHCO_3$ aqueous solution and extracted with EtOAc (20 mL x 3). The organic layer was dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. The resultant residue was treated with 1 M aqueous solution of HCl (10 mL) in THF (10 mL) for 30 min at room temperature. This reaction mixture was diluted with H_2O (10 mL), extracted with EtOAc (20 mL x 3), dried over anhydrous $MgSO_4$ and evaporated. Purification of the obtained residue by column chromatography on silica gel (hexane/EtOAc = 2 : 1) gave an inseparable mixture of *anti*-3fa and *syn*-3fa (107 mg, 0.47 mmol, 93% yield, *anti/syn* = 5.0 : 1). Colorless oil; IR (neat) ν 3031, 2919, 1756, 1715, 1602, 1496, 1163, 1454, 767, 703 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$) δ for *anti*-3fa 2.06 (3H, s), 2.90 (1H, dd, $J = 17.5$, 8.1 Hz), 3.03 (1H, dd, $J = 17.5$, 5.3 Hz), 3.41–3.48 (1H, m), 5.15 (1H, dt, $J = 7.5$, 1.9 Hz), 6.09 (1H, dd, $J = 5.7$, 1.9

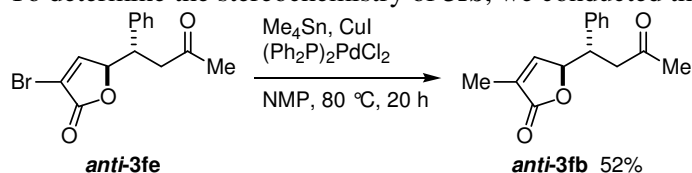
Hz), 7.09-7.35 (6H, m), for **syn-3fa** 2.17 (3H, s), 2.86-2.96 (1H, m), 3.23 (1H, dd, $J = 18.2, 8.3$ Hz), 3.69-3.76 (1H, m), 5.34 (1H, quin, $J = 1.9$ Hz), 7.09-7.35 (6H, m); ^{13}C NMR (100 MHz, CDCl_3) δ for **anti-3fa** 30.5, 44.3, 45.0, 85.7, 122.0, 127.8, 128.1, 129.0, 139.4, 155.4, 172.6, 205.8, for **syn-3fa** 30.5, 42.7, 44.8, 84.2, 122.2, 127.7, 128.3, 128.7, 137.0, 155.1, 172.8, 206.5; MS (ESI-TOF) m/z 253 $[\text{M}+\text{Na}]^+$; HRMS calcd for $\text{C}_{14}\text{H}_{14}\text{NaO}_3$ $[\text{M}+\text{Na}]^+$, 253.0841; found, 253.0821. Anal. Calcd for $\text{C}_{14}\text{H}_{14}\text{O}_3$: C, 73.08; H, 6.13. Found: C, 72.80; H, 6.21.

(*R)-3-Methyl-5-[(*S**)-3-oxo-1-phenylbutyl]furan-2(5*H*)-one (*anti*-3fb) and (*S**)-3-methyl-5-[(*S**)-3-oxo-1-phenylbutyl]furan-2(5*H*)-one (*syn*-3fb)**



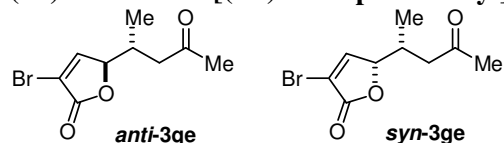
According to the synthetic procedure for **3fa**, 1,4-adduct **3fb** was obtained in 87% yield (**anti-3fb**, 88.2 mg, 0.36 mmol; **syn-3fb**, 0.07 mmol, 18.0 mg) by the reaction of **2f** (73 mg, 0.50 mmol) with *tert*-butyl(dimethyl)[(3-methylfuran-2-yl)oxy]silane (127 mg, 0.60 mmol) in the presence of carbon acid **1** (2.9 mg, 5.0 μmol) in CH_2Cl_2 (1.0 mL) at -78°C for 2 h. For **anti-3fb** Colorless crystals; Mp. $60.1\text{--}61.0^\circ\text{C}$; IR (KBr) ν 3030, 3006, 1752, 1711, 1498, 1456, 1163, 768, 703 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.86 (3H, brs), 2.03 (3H, s), 2.87 (1H, dd, $J = 17.4, 8.4$ Hz), 2.99 (1H, dd, $J = 17.4, 5.0$ Hz), 3.30-3.41 (1H, m), 4.98 (1H, dt, $J = 7.6, 1.6$ Hz), 6.77 (1H, brs), 7.15-7.28 (3H, m), 7.28-7.36 (2H, m); ^{13}C NMR (100 MHz, CDCl_3) δ 10.6, 30.5, 44.6, 45.2, 83.4, 127.6, 128.0, 128.9, 130.5, 139.6, 147.7, 173.7, 205.9; MS (ESI-TOF) m/z 245 $[\text{M}+\text{H}]^+$; HRMS calcd for $\text{C}_{15}\text{H}_{17}\text{O}_3$ $[\text{M}+\text{H}]^+$, 245.1178; found, 245.1189. For **syn-3fb** Colorless oil; IR (neat) ν 3031, 1757, 1714, 1496, 1454, 1162, 763, 703 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.67 (3H, brs), 2.14 (3H, s), 2.89 (1H, dd, $J = 18.0, 6.2$ Hz), 3.16 (1H, dd, $J = 18.0, 7.9$ Hz), 3.68 (1H, ddd, $J = 7.9, 6.2, 3.4$ Hz), 5.15 (1H, dt, $J = 3.4, 1.7$ Hz), 6.82-6.88 (1H, m), 7.07-7.14 (2H, m), 7.15-7.28 (3H, m); ^{13}C NMR (100 MHz, CDCl_3) δ 10.4, 30.5, 42.9, 44.6, 82.0, 127.5, 128.3, 128.6, 130.8, 137.3, 147.3, 173.9, 206.6; MS (ESI-TOF) m/z 245 $[\text{M}+\text{H}]^+$; HRMS calcd for $\text{C}_{15}\text{H}_{17}\text{O}_3$ $[\text{M}+\text{H}]^+$, 245.1178; found, 245.1188.

To determine the stereochemistry of **3fb**, we conducted the following methylation reaction.



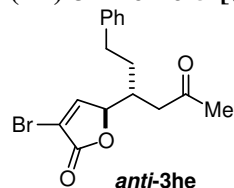
To a solution of (*R**)-3-bromo-5-[(*S**)-3-oxo-1-phenylbutyl]furan-2(5*H*)-one **anti-3fe** (88 mg, 0.29 mmol) in *N*-methyl-2-pyrrolidone (NMP, 2.0 mL), $(\text{Ph}_3\text{P})_2\text{PdCl}_2$ (30 mg, 0.04 mmol), CuI (34 mg, 0.17 mmol) and Me_4Sn (0.16 mL, 1.2 mmol) were added. After being stirred for 20 h at 80°C , the resultant mixture was quenched with H_2O (10 mL), extracted with EtOAc (10 mL x 3) and dried over anhydrous MgSO_4 . After the concentration of this organic layer under reduced pressure, the residue was purified by column chromatography on silica gel (hexane/EtOAc = 2 : 1) to give (*R**)-3-methyl-5-[(*S**)-3-oxo-1-phenylbutyl]furan-2(5*H*)-one **anti-3fb** in 52% yield (35.0 mg, 0.15 mmol) without the formation of **syn-3fb**.

(R*)-3-Bromo-5-[(R*)-4-oxopentan-2-yl]furan-2(5H)-one (*anti*-3ge) and (S*)-3-bromo-5-[(R*)-4-oxopentan-2-yl]furan-2(5H)-one (*syn*-3ge)



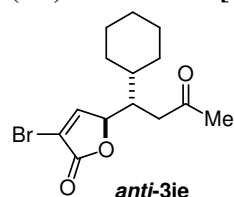
According to the synthetic procedure for **3fa**, an inseparable mixture of *anti*-3ge and *syn*-3ge was obtained in 76% yield (94 mg, 0.38 mmol, *anti*/*syn* = 12 : 1) by the reaction of α,β -enone **2g** (42 mg, 0.50 mmol) with (3-bromofuran-2-yloxy)triethylsilane (166 mg, 0.60 mmol) in the presence of carbon acid **1** (1.4 mg, 2.5 μ mol) in CH_2Cl_2 (1.0 mL) at -78°C for 2 h. Colorless oil; IR (neat) ν 3088, 2970, 1770, 1713, 1607, 1369, 1160, 981 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ for *anti*-3ge 1.04 (3H, m), 2.09 (3H, m), 2.33 (1H, ddd, J = 16.7, 6.7, 2.6 Hz), 2.38-2.58 (2H, m), 4.89 (1H, dt, J = 5.4, 2.0 Hz), 7.49 (1H, brd, J = 2.0 Hz), for *syn*-3ge 0.80 (3H, dd, J = 6.9, 3.5 Hz), 2.13 (3H, d, J = 3.9 Hz), 2.38-2.58 (2H, m), 2.59 (1H, ddd, J = 17.7, 6.9, 4.6 Hz), 5.12-5.30 (1H, m), 7.47 (1H, d, J = 1.6 Hz); ^{13}C NMR (100 MHz, CDCl_3) δ for *anti*-3ge 16.4, 29.6, 32.1, 44.5, 85.7, 113.2, 151.9, 168.0, 206.4, for *syn*-3ge 13.4, 30.4, 31.1, 45.9, 84.7, 113.2, 151.9, 168.3, 206.7; MS (ESI-TOF) m/z 269 $[\text{M}+\text{Na}]^+$, 271 $[\text{M}+2+\text{Na}]^+$; HRMS calcd for $\text{C}_9\text{H}_{11}\text{BrNaO}_3$ $[\text{M}+\text{Na}]^+$, 268.9789; found, 268.9792; Anal. Calcd for $\text{C}_9\text{H}_{11}\text{BrO}_3$: C, 43.75; H, 4.49. Found: C, 43.62; H, 4.59.

(R*)-3-Bromo-5-[(R*)-5-oxo-1-phenylhexan-3-yl]furan-2(5H)-one (*anti*-3he)



According to the synthetic procedure for **3fa**, 1,4-adduct *anti*-3he was obtained in 91% yield (153 mg, 0.45 mmol) by the reaction of α,β -enone **2c** (87 mg, 0.50 mmol) with (3-bromofuran-2-yloxy)triethylsilane (166 mg, 0.60 mmol) in the presence of carbon acid **1** (1.4 mg, 2.5 μ mol) in CH_2Cl_2 (1.0 mL) at -78°C for 2 h. Colorless oil; IR (neat) ν 3086, 3026, 1772, 1713, 1604, 1496, 1454, 1155, 984, 752, 704 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.61-1.72 (1H, m), 1.72-1.86 (1H, m), 2.01 (3H, s), 2.22-2.36 (2H, m), 2.41-2.51 (1H, m), 2.54-2.68 (2H, m), 5.01 (1H, dd, J = 3.8, 1.7 Hz), 7.06-7.16 (3H, m), 7.17-7.25 (2H, m), 7.33 (1H, d, J = 1.7 Hz); ^{13}C NMR (100 MHz, CDCl_3) δ 30.4, 33.0, 33.3, 35.9, 41.6, 84.0, 112.8, 126.2, 128.2, 128.6, 140.8, 152.5, 168.2, 206.7; MS (ESI-TOF) m/z 337 $[\text{M}+\text{H}]^+$, 339 $[\text{M}+2+\text{H}]^+$; HRMS calcd for $\text{C}_{16}\text{H}_{18}\text{BrO}_3$ $[\text{M}+\text{H}]^+$, 337.0439; found, 337.0449; Anal. Calcd for $\text{C}_{16}\text{H}_{17}\text{BrO}_3$: C, 56.99; H, 5.08. Found: C, 56.89; H, 5.06.

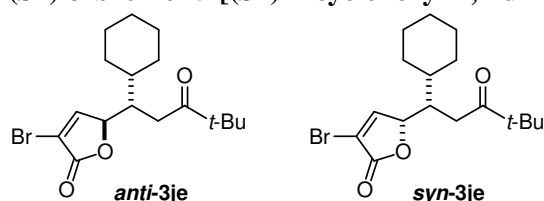
(R*)-3-Bromo-5-[(S*)-1-cyclohexyl-3-oxobutyl]furan-2(5H)-one (*anti*-3ie)



According to the synthetic procedure for **3fa**, 1,4-adduct *anti*-3ie was obtained in 93% yield (146 mg, 0.47 mmol) by the reaction of α,β -enone **2i** (76 mg, 0.50 mmol) with (3-bromofuran-2-yloxy)triethylsilane (166 mg, 0.60

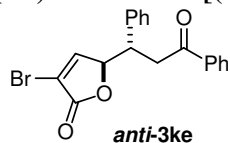
mmol) in the presence of carbon acid **1** (0.71 mg, 1.3 μ mol) in CH_2Cl_2 (1.0 mL) at -78°C for 2 h. The stereochemistry of **anti-3ie** was determined by an X-ray crystallographic analysis. Colorless crystals; Mp. 105.0-105.3 $^\circ\text{C}$; IR (KBr) ν 3102, 2930, 2847, 1758, 1716, 1607, 992 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 0.91-1.27 (5H, m), 1.36-1.48 (1H, m), 1.58-1.82 (5H, m), 2.06 (3H, s), 2.17 (1H, dd, $J = 18.1, 7.0$ Hz), 2.33, (1H, dd, $J = 18.1, 4.1$ Hz), 2.34-2.41 (1H, m), 5.16 (1H, brs), 7.37 (1H, d, $J = 1.5$ Hz); ^{13}C NMR (100 MHz, CDCl_3) δ 26.1, 26.3, 30.3, 30.6, 30.8, 39.1, 40.0, 40.8, 83.1, 112.1, 153.8, 168.4, 207.1; MS (ESI-TOF) m/z 337 $[\text{M}+\text{Na}]^+$, 339 $[\text{M}+2+\text{Na}]^+$; HRMS calcd for $\text{C}_{14}\text{H}_{19}\text{BrNaO}_3$ $[\text{M}+\text{Na}]^+$, 337.0415; found, 337.0405; Anal. Calcd for $\text{C}_{14}\text{H}_{19}\text{BrO}_3$: C, 53.35; H, 6.08. Found: C, 53.20; H, 6.19.

(*R)-3-Bromo-5-[(*S**)-1-cyclohexyl-4,4-dimethyl-3-oxopentyl]furan-2(5*H*)-one (*anti*-3je) and (*S**)-3-bromo- 5-[(*S**)-1-cyclohexyl-4,4-dimethyl-3-oxopentyl]furan-2(5*H*)-one (*syn*-3je)**



According to the synthetic procedure for **3fa**, 1,4-adduct **3je** was obtained in 66% yield (**anti-3je**, 106 mg, 0.30 mmol; **syn-3je**, 12 mg, 0.04 mmol) by the reaction of α,β -enone **2j** (97 mg, 0.50 mmol) with (3-bromofuran-2-yloxy)triethylsilane (166 mg, 0.60 mmol) in the presence of carbon acid **1** (2.8 mg, 5.0 μ mol) in CH_2Cl_2 (1.0 mL) at -24°C for 2 h. The stereochemistry of **3je** was determined by an X-ray crystallographic analysis of **anti-3je**. For **anti-3je** Colorless crystals; Mp. 108.8-109.3 $^\circ\text{C}$; IR (KBr) ν 3113, 2928, 1764, 1700, 1609, 988 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 0.50-1.29 (5H, m), 1.07 (9H, s), 1.36-1.48 (1H, m), 1.60-1.85 (5H, m), 2.33 (2H, d, $J = 5.6$ Hz), 2.42-2.49 (1H, m), 5.19 (1H, t, $J = 1.6$ Hz), 7.37 (1H, d, $J = 1.6$ Hz); ^{13}C NMR (100 MHz, CDCl_3) δ 26.2, 26.4, 26.5, 26.6, 30.6, 31.1, 32.3, 40.1, 40.6, 44.3, 83.2, 111.7, 154.2, 168.7, 214.6; MS (ESI-TOF) m/z 357 $[\text{M}+\text{H}]^+$, 359 $[\text{M}+2+\text{H}]^+$; HRMS calcd for $\text{C}_{17}\text{H}_{26}\text{BrO}_3$ $[\text{M}+\text{H}]^+$, 357.1065; found, 357.1059; Anal. Calcd for $\text{C}_{17}\text{H}_{25}\text{BrO}_3$: C, 57.15; H, 7.05. Found: C, 57.11; H, 7.00. For **syn-3je** Colorless crystals; Mp. 64.5-65.0 $^\circ\text{C}$; IR (KBr) ν 3120, 2923, 2852, 1766, 1697, 1612, 1000 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 0.94-1.32 (5H, m), 1.16 (9H, s), 1.38-1.48 (1H, m), 1.51-1.78 (5H, m), 2.37-2.44 (1H, m), 2.52 (1H, dd, $J = 18.4, 4.4$ Hz), 2.65 (1H, dd, $J = 18.4, 7.8$ Hz), 5.08 (1H, dd, $J = 5.0, 1.7$ Hz), 7.49 (1H, d, $J = 1.7$ Hz); ^{13}C NMR (100 MHz, CDCl_3) δ 26.3, 26.5, 26.58, 26.63, 30.1, 31.8, 33.5, 38.3, 41.3, 44.6, 84.4, 112.9, 152.6, 168.4, 214.5; MS (ESI-TOF) m/z 357 $[\text{M}+\text{H}]^+$, 359 $[\text{M}+2+\text{H}]^+$; HRMS calcd for $\text{C}_{17}\text{H}_{26}\text{BrO}_3$ $[\text{M}+\text{H}]^+$, 357.1065; found, 357.1073.

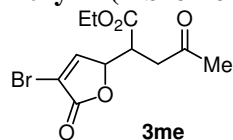
(*R)-3-Bromo-5-[(*S**)-3-oxo-1,3-diphenylpropyl]furan-2(5*H*)-one (*anti*-3ke)**



According to the procedure of **3fa**, the reaction of α,β -enone **2k** (97 mg, 0.50 mmol) with (3-bromofuran-2-yloxy)triethylsilane (166 mg, 0.60 mmol) in the presence of carbon acid **1** (2.8 mg, 5.0 μ mol) was conducted at -78°C

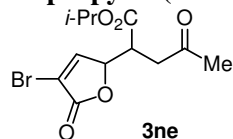
for 2 h. After extractive work up, the resultant mixture was treated with triflic acid in CH_2Cl_2 at -78°C for 1 h. Then, this reaction mixture was quenched with H_2O (50 mL), extracted with CH_2Cl_2 (40 mL), dried over MgSO_4 and concentrated. The resulting residue was purified by column chromatography on silica gel (hexane/EtOAc = 4 : 1) to give 1,4-adduct **anti-3ke** in 95% yield (176 mg, 0.48 mmol, *anti/syn* = >30 : 1). Colorless crystals; Mp. $34.0\text{--}34.6^\circ\text{C}$; IR (KBr) ν 3087, 2914, 1774, 1685, 1598, 1496, 1449, 993, 752, 727, 700 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 3.42 (1H, dd, $J = 17.6, 8.0\text{ Hz}$), 3.50 (1H, dd, $J = 17.6, 5.2\text{ Hz}$), 3.66 (1H, td, $J = 7.8, 5.2\text{ Hz}$), 5.15 (1H, dd, $J = 7.8, 1.6\text{ Hz}$), 7.18–7.31 (6H, m), 7.32–7.40 (2H, m), 7.47 (1H, t, $J = 7.4\text{ Hz}$), 7.82 (2H, d, $J = 7.4\text{ Hz}$); ^{13}C NMR (100 MHz, CDCl_3) δ 39.9, 44.4, 85.0, 113.5, 127.8, 127.9, 128.1, 128.6, 129.0, 133.3, 136.5, 138.8, 152.0, 167.9, 197.0; MS (ESI-TOF) m/z 371 $[\text{M}+\text{H}]^+$, 373 $[\text{M}+2+\text{H}]^+$; HRMS calcd for $\text{C}_{19}\text{H}_{16}\text{BrO}_3$ $[\text{M}+\text{H}]^+$, 371.0283; found, 371.0277; Anal. Calcd for $\text{C}_{19}\text{H}_{15}\text{BrO}_3$: C, 61.47; H, 4.07. Found: C, 61.50; H, 4.20.

Ethyl 2-(4-bromo-5-oxo-2,5-dihydrofuran-2-yl)-4-oxopentanoate (**3me**)



According to the synthetic procedure for **3fa**, 1,4-adduct **3me** was obtained in 96% yield (**3me-major**, 124 mg, 0.41 mmol; **3me-minor**, 22 mg, 0.07 mmol) by the reaction of α,β -enone **2m** (71 mg, 0.50 mmol) with (3-bromofuran-2-yloxy)triethylsilane (166 mg, 0.60 mmol) in the presence of carbon acid **1** (1.4 mg, 2.5 μmol) in CH_2Cl_2 (1.0 mL) at -78°C for 2 h. For **3me-major** Colorless oil; IR (neat) ν 3093, 2983, 1779, 1721, 1608, 1370, 1188, 1163, 984 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.26 (3H, t, $J = 7.1\text{ Hz}$), 2.14 (3H, s), 2.60 (1H, dd, $J = 18.3, 4.9\text{ Hz}$), 3.02 (1H, dd, $J = 18.3, 6.8\text{ Hz}$), 3.14–3.21 (1H, m), 4.19 (2H, q, $J = 7.1\text{ Hz}$), 5.39 (1H, dd, $J = 6.2, 1.8\text{ Hz}$), 7.61 (1H, d, $J = 1.8\text{ Hz}$); ^{13}C NMR (100 MHz, CDCl_3) δ 14.0, 29.9, 39.1, 43.0, 61.8, 81.1, 113.8, 151.6, 167.4, 169.4, 205.3; MS (ESI-TOF) m/z 327 $[\text{M}+\text{Na}]^+$, 329 $[\text{M}+2+\text{Na}]^+$; HRMS calcd for $\text{C}_{11}\text{H}_{13}\text{BrNaO}_5$ $[\text{M}+\text{Na}]^+$, 326.9844; found, 326.9849; Anal. Calcd for $\text{C}_{11}\text{H}_{13}\text{BrO}_5$: C, 43.30; H, 4.29. Found: C, 43.22; H, 4.35. For **3me-minor** Colorless oil; IR (neat) ν 3094, 2983, 1776, 1720, 1608, 1371, 1162, 1026, 985 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.24 (3H, t, $J = 7.1\text{ Hz}$), 2.18 (3H, s), 2.68 (1H, dd, $J = 18.3, 5.3\text{ Hz}$), 2.89 (1H, dd, $J = 18.3, 7.6\text{ Hz}$), 3.43–3.55 (1H, m), 4.06–4.24 (2H, m), 5.28 (1H, dd, $J = 4.2, 1.7\text{ Hz}$), 7.54 (1H, d, $J = 1.7\text{ Hz}$); ^{13}C NMR (100 MHz, CDCl_3) δ 14.1, 29.9, 39.5, 42.6, 61.7, 80.8, 113.4, 151.1, 167.5, 169.6, 205.2; MS (ESI-TOF) m/z 305 $[\text{M}+\text{H}]^+$, 307 $[\text{M}+2+\text{H}]^+$; HRMS calcd for $\text{C}_{11}\text{H}_{13}\text{BrO}_5$ $[\text{M}+\text{H}]^+$, 305.0025; found, 305.0021.

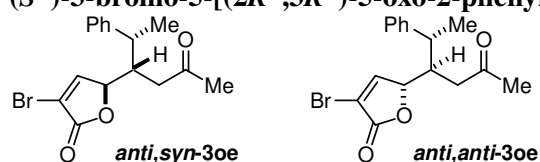
Isopropyl 2-(4-bromo-5-oxo-2,5-dihydrofuran-2-yl)-4-oxopentanoate (**3ne**)



According to the synthetic procedure for **3fa**, 1,4-adduct **3ne** was obtained in 80% yield (**3ne-major**, 111 mg, 0.35 mmol; **3ne-minor**, 17 mg, 0.05 mmol) by the reaction of α,β -enone **2n** (78 mg, 0.50 mmol) with (3-bromofuran-2-yloxy)triethylsilane (166 mg, 0.60 mmol) in the presence of carbon acid **1** (2.8 mg, 5.0 μmol) in CH_2Cl_2 (1.0 mL) at -78°C for 2 h. For **3ne-major** Colorless oil; IR (neat) ν 3092, 2982, 1780, 1721, 1608, 1374, 1107, 984 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.248 (3H, d, $J = 6.3\text{ Hz}$), 1.252 (3H, d, $J = 6.3\text{ Hz}$), 2.18

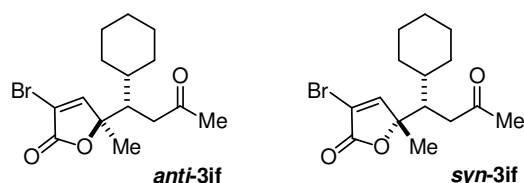
(3H, s), 2.60 (1H, dd, $J = 18.2, 4.9$ Hz), 3.03 (1H, dd, $J = 18.2, 6.9$ Hz), 3.05-3.14 (1H, m), 5.06 (1H, septet, $J = 6.3$ Hz), 5.38 (1H, dd, $J = 6.2, 1.7$ Hz), 7.61 (1H, d, $J = 1.7$ Hz); ^{13}C NMR (100 MHz, CDCl_3) δ 21.5, 29.9, 39.1, 43.1, 69.5, 81.3, 113.6, 151.7, 167.5, 169.4, 205.3; MS (ESI-TOF) m/z 341 $[\text{M}+\text{Na}]^+$, 343 $[\text{M}+2+\text{Na}]^+$; HRMS calcd for $\text{C}_{12}\text{H}_{15}\text{BrNaO}_5$ $[\text{M}+\text{Na}]^+$, 341.0001; found, 340.9983; Anal. Calcd for $\text{C}_{12}\text{H}_{15}\text{BrO}_5$: C, 45.16; H, 4.74. Found: C, 45.45; H, 4.97. For **3ne-minor** Colorless oil; IR (neat) ν 3095, 2982, 1779, 1721, 1609, 1375, 1166, 1107, 984 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.21 (3H, d, $J = 6.3$ Hz), 1.22 (3H, d, $J = 6.3$ Hz), 2.18 (3H, s), 2.68 (1H, dd, $J = 18.3, 5.5$ Hz), 2.91 (1H, dd, $J = 18.3, 7.6$ Hz), 3.40-3.45 (1H, m), 5.00 (1H, septet, $J = 6.3$ Hz), 5.26 (1H, dd, $J = 4.0, 1.7$ Hz), 7.54 (1H, d, $J = 1.7$ Hz); ^{13}C NMR (100 MHz, CDCl_3) δ 21.66 and 21.73, 30.0, 39.8, 42.9, 69.5, 81.0, 113.4, 151.3, 167.6, 169.0, 205.3; MS (ESI-TOF) m/z 341 $[\text{M}+\text{Na}]^+$, 343 $[\text{M}+2+\text{Na}]^+$; HRMS calcd for $\text{C}_{12}\text{H}_{15}\text{BrNaO}_5$ $[\text{M}+\text{Na}]^+$, 341.0001; found, 340.9997.

(*R)-3-Bromo-5-[(2*R**,3*S**)-5-oxo-2-phenylhexan-3-yl]furan-2(5*H*)-one (*anti,syn*-3oe) and (*S**)-3-bromo-5-[(2*R**,3*R**)-5-oxo-2-phenylhexan-3-yl]furan-2(5*H*)-one (*anti,anti*-3oe)**



According to the synthetic procedure for **3fa**, 1,4-adduct **3oe** was obtained in 91% yield (**anti,syn-3oe** 109 mg, 0.33 mmol; **anti,anti-3oe** 44 mg, 0.13 mmol) by the reaction of **2o** (87 mg, 0.50 mmol) with (3-bromofuran-2-yloxy)triethylsilane (166 mg, 0.60 mmol) in the presence of carbon acid **1** (0.71 mg, 1.3 μmol) in CH_2Cl_2 (1.0 mL) at -78°C for 2 h. The stereochemistry of **3oe** was determined by an X-ray crystallographic analysis of **anti,anti-3oe**. For **anti,syn-3oe** Colorless crystals; Mp. $86.8\text{--}87.1^\circ\text{C}$; IR (KBr) ν 3102, 3032, 1758, 1713, 1606, 1454, 1424, 1168, 1032, 982, 768, 701 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.25 (3H, d, $J = 6.9$ Hz), 2.13 (3H, s), 2.25 (1H, dd, $J = 18.8, 7.0$ Hz), 2.50 (1H, dd, $J = 18.8, 3.7$ Hz), 2.72-2.80 (1H, m), 2.82 (1H, d of quintet, $J = 10.7, 6.9$ Hz), 4.71 (1H, t, $J = 1.7$ Hz), 7.18 (1H, d, $J = 1.7$ Hz), 7.21-7.36 (5H, m); ^{13}C NMR (100 MHz, CDCl_3) δ 20.0, 30.4, 39.2, 41.5, 42.3, 83.2, 111.9, 127.0, 127.6, 128.9, 144.3, 153.7, 168.4, 207.0; MS (ESI-TOF) m/z 359 $[\text{M}+\text{Na}]^+$, 361 $[\text{M}+2+\text{Na}]^+$; HRMS calcd for $\text{C}_{16}\text{H}_{17}\text{BrNaO}_3$ $[\text{M}+\text{Na}]^+$, 359.0259; found, 359.0253; Anal. Calcd for $\text{C}_{16}\text{H}_{17}\text{BrO}_3$: C, 56.99; H, 5.08. Found: C, 56.97; H, 5.14. For **anti,anti-3oe** Colorless crystals; Mp. $69.8\text{--}70.0^\circ\text{C}$; IR (KBr) ν 3111, 3027, 2966, 1771, 1710, 1605, 1492, 1454, 1164, 1056, 982, 765, 703 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.41 (3H, d, $J = 6.9$ Hz), 1.85 (3H, s), 2.12 (2H, d, $J = 5.7$ Hz), 2.80-2.88 (1H, m), 2.88-2.98 (1H, m), 5.30 (1H, brs), 7.17 (2H, d, $J = 7.3$ Hz), 7.30 (1H, t, $J = 7.3$ Hz), 7.37 (1H, d, $J = 1.6$ Hz); ^{13}C NMR (100 MHz, CDCl_3) δ 19.5, 30.3, 39.9, 41.3, 41.7, 82.1, 112.2, 126.9, 127.6, 128.9, 144.5, 153.7, 168.5, 207.2; MS (ESI-TOF) m/z 359 $[\text{M}+\text{Na}]^+$, 361 $[\text{M}+2+\text{Na}]^+$; HRMS calcd for $\text{C}_{16}\text{H}_{17}\text{BrNaO}_3$ $[\text{M}+\text{Na}]^+$, 359.0259; found, 359.0243.

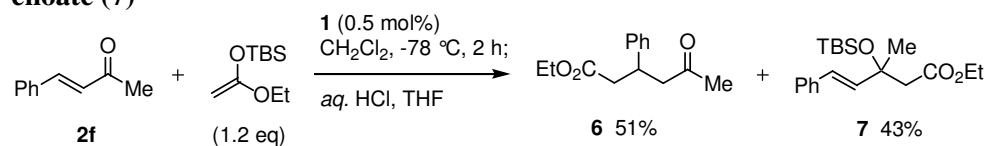
(*R)-3-Bromo-5-[(*S**)-1-cyclohexyl-3-oxobutyl]-5-methylfuran-2(5*H*)-one (*anti*-3if) and (*S**)-3-bromo-5-[(*S**)-1-cyclohexyl-3-oxobutyl]-5-methylfuran-2(5*H*)-one (*syn*-3if)**



According to the synthetic procedure for **3fa**, 1,4-adduct **3if** was obtained in 97% yield (**anti-3if**, 139 mg, 0.43 mmol; **syn-3if**, 21 mg, 0.06 mmol) by the reaction of α,β -enone **2i** (76 mg, 0.50 mmol) with (3-bromo-5-methylfuran-2-yloxy)triethylsilane (175 mg, 0.60 mmol) in the presence of carbon acid **1** (0.71 mg, 1.3 μ mol) in CH_2Cl_2 (1.0 mL) at -78°C for 2 h. The stereochemistry of **3if** was determined by an X-ray crystallographic analysis of **anti-3if**. For **anti-3if** Colorless crystals; Mp. $79.0\text{--}79.6^\circ\text{C}$; IR (KBr) ν 3103, 2997, 2920, 2852, 1775, 1703, 1612, 992 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 0.83 (1H, qd, $J = 13.1, 3.4$ Hz), 0.95–1.08 (1H, m), 1.17 (1H, qt, $J = 12.9, 3.4$ Hz), 1.27 (1H, qt, $J = 12.9, 3.4$ Hz), 1.43 (1H, d, $J = 12.8$ Hz), 1.48 (3H, s), 1.58–1.80 (3H, m), 1.61 (1H, d, $J = 12.8$ Hz), 1.85 (1H, d, $J = 13.1$ Hz), 2.05 (3H, s), 2.14 (1H, dd, $J = 18.7, 8.2$ Hz), 2.30 (1H, dd, $J = 18.7, 3.0$ Hz), 2.36 (1H, dt, $J = 8.2, 3.0$ Hz), 7.27 (1H, brs); ^{13}C NMR (100 MHz, CDCl_3) δ 22.2, 26.1, 26.2, 26.7, 28.2, 30.3, 32.7, 37.4, 38.6, 43.9, 90.9, 110.8, 158.8, 168.0, 207.4; MS (ESI-TOF) m/z 329 $[\text{M}+\text{H}]^+$, 331 $[\text{M}+2+\text{H}]^+$; HRMS calcd for $\text{C}_{15}\text{H}_{22}\text{BrO}_3$ $[\text{M}+\text{H}]^+$, 329.0752; found, 329.0772; Anal. Calcd for $\text{C}_{15}\text{H}_{21}\text{BrO}_3$: C, 54.72; H, 6.43. Found: C, 54.73; H, 6.40. For **syn-3if** Colorless crystals; Mp. $78.8\text{--}79.3^\circ\text{C}$; IR (KBr) ν 3087, 2928, 2852, 1766, 1716, 1607, 988 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 0.84–1.31 (5H, m), 1.35–1.47 (2H, m), 1.41 (3H, s), 1.47–1.59 (1H, m), 1.69 (1H, brd, $J = 12.6$ Hz), 1.67–1.76 (2H, m), 2.19 (3H, s), 2.41 (1H, dd, $J = 18.3, 4.9$ Hz), 2.47 (1H, dd, $J = 18.3, 6.0$ Hz), 2.53–2.59 (1H, m), 7.39 (1H, brs); ^{13}C NMR (100 MHz, CDCl_3) δ 24.5, 26.1, 26.4, 26.8, 28.8, 30.1, 32.9, 39.2, 39.8, 45.2, 91.7, 112.0, 156.5, 167.6, 206.7; MS (ESI-TOF) m/z 329 $[\text{M}+\text{H}]^+$, 331 $[\text{M}+2+\text{H}]^+$; HRMS calcd for $\text{C}_{15}\text{H}_{22}\text{BrO}_3$ $[\text{M}+\text{H}]^+$, 329.0752; found, 329.0768; Anal. Calcd for $\text{C}_{15}\text{H}_{21}\text{BrO}_3$: C, 54.72; H, 6.43. Found: C, 54.54; H, 6.36.

3.3. Reaction of benzalacetone (**2f**) with acyclic ketene silyl acetal

Ethyl 5-oxo-3-phenylhexanoate (6) and ethyl (*E*)-3-(*tert*-butyldimethylsilyloxy)-3-methyl-5-phenylpent-4-enoate (**7**)

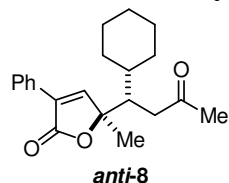


According to the synthetic procedure for **3fa**, 1,4-adduct **6** and 1,2-adduct **7** were obtained in 51% yield (60 mg, 0.26 mmol) and 43% yield (75 mg, 0.22 mmol), respectively, by the reaction of **2f** (73 mg, 0.50 mmol) with *tert*-butyl(1-ethoxyvinyl)oxydimethylsilane (151 mg, 0.75 mmol) in the presence of carbon acid **1** (0.71 mg, 1.3 μ mol) in CH_2Cl_2 (1.0 mL) at -78°C for 2 h. The structure of 1,4-adduct **6** was confirmed by comparison to the reported spectrum data.¹⁷ For 1,2-adduct **7** Colorless oil; IR (neat) ν 3026, 2956, 2930, 2895, 2856, 1736, 1495, 1472, 971, 775, 693 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 0.09 (3H, s), 0.10, (3H, s), 0.91 (9H, s), 1.23 (3H, t, $J = 7.1$ Hz), 1.60 (3H, s), 2.60 (1H, d, $J = 13.4$ Hz), 2.64 (1H, d, $J = 13.4$ Hz), 4.10 (2H, q, $J = 7.1$ Hz), 6.42 (1H, d, $J = 16.1$ Hz), 6.55 (1H, d, $J = 16.1$ Hz), 7.21 (1H, t, $J = 7.3$ Hz), 7.32 (2H, t, $J = 7.3$ Hz), 7.39 (2H, d, $J = 7.3$ Hz); ^{13}C NMR (100 MHz, CDCl_3) δ –2.2 and –2.1, 14.2, 18.2, 25.8, 27.4, 49.4, 60.2, 74.0, 126.5, 127.45, 127.47, 128.6, 136.1, 137.0, 170.5; MS (ESI-TOF) m/z 371 $[\text{M}+\text{Na}]^+$; HRMS calcd for $\text{C}_{20}\text{H}_{32}\text{BrNaO}_3\text{Si}$ $[\text{M}+\text{Na}]^+$,

371.2018; found, 371.2009; Anal. Calcd for C₂₀H₃₂O₃Si: C, 68.92; H, 9.25. Found: C, 68.64; H, 9.26.

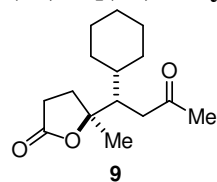
4. Some transformations of α -bromo- γ -butenolides

(*R**)-5-[(*S**)-1-Cyclohexyl-3-oxobutyl]-5-methyl-3-phenylfuran-2(5*H*)-one (*anti*-8)



To a solution of ***anti*-3if** (33 mg, 0.10 mmol) and (Ph₃P)₄Pd (6 mg, 3.5 μ mol) in a mixed solvent of 1,2-dimethoxyethane-EtOH (5 : 1, 0.6 mL), phenylboronic acid (12 mg, 0.10 mmol) and 2.0 M aqueous solution of Na₂CO₃ (0.4 mL, 0.80 mmol) were added at room temperature. After being refluxed for 1 h, the reaction mixture was quenched with H₂O (10 mL) and extracted with EtOAc (10 mL x 3). The combined organic layer was washed with brine (10 mL), dried over anhydrous MgSO₄ and evaporated. The obtained residue was purified by column chromatography on silica gel (hexane/EtOAc = 5 : 1) to give coupling product ***anti*-8** in 95% yield (31 mg, 0.10 mmol). Colorless crystals; Mp. 129.5-131.1 °C; IR (KBr) ν 3109, 3073, 2993, 2925, 2854, 1746, 1708, 1495, 1451, 1415, 795, 693 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 0.90 (1H, qd, *J* = 12.6, 3.4 Hz), 1.01-1.16 (1H, m), 1.24 (1H, qt, *J* = 13.0, 3.5 Hz), 1.34 (1H, qt, *J* = 13.0, 3.5 Hz), 1.51 (1H, brd, *J* = 12.6 Hz), 1.56 (3H, s), 1.67 (1H, brd, *J* = 12.6 Hz), 1.76 (2H, brd, *J* = 12.6 Hz), 1.81-1.93 (1H, m), 1.95 (3H, s), 2.02 (1H, d, *J* = 13.5 Hz), 2.25 (1H, dd, *J* = 18.7, 8.0 Hz), 2.33 (1H, dd, *J* = 18.7, 3.3 Hz), 2.47 (1H, dt, *J* = 8.0, 3.3 Hz), 7.32-7.42 (4H, m), 7.75 (1H, d, *J* = 1.5 Hz), 7.77 (1H, d, *J* = 1.9 Hz); ¹³C NMR (100 MHz, CDCl₃) δ 22.6, 26.3, 26.4, 26.9, 28.3, 30.5, 32.9, 37.6, 38.8, 44.1, 87.8, 126.9, 128.7, 129.1, 129.2, 154.5, 171.5, 208.0; MS (ESI-TOF) *m/z* 327 [M+H]⁺; HRMS calcd for C₂₁H₂₆O₃ [M+H]⁺, 327.1936; found, 327.1945; Anal. Calcd for C₂₁H₂₆O₃: C, 77.27; H, 8.03. Found: C, 77.10; H, 7.94.

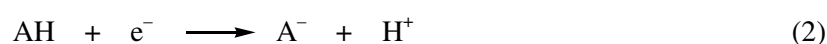
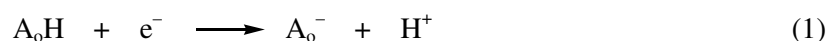
(*S**)-5-[(*S**)-1-Cyclohexyl-3-oxobutyl]-5-methyldihydrofuran-2(3*H*)-one (**9**)



In the presence of 10% palladium on carbon (10 mg, 0.01 mmol), a solution of ***anti*-3if** (33 mg, 0.10 mmol) in EtOH (2.5 mL) was stirred at room temperature for 3 h under H₂ atmosphere. The resultant mixture was filtrated through celite pad. The filtrate was evaporated and purified by column chromatography on silica gel (hexane/EtOAc = 1 : 1) to give γ -butanolide **9** in 99% yield (25.0 mg, 0.099 mmol). Colorless crystals; Mp. 70.5-71.5 °C; IR (KBr) ν 2963, 2922, 2853, 1758, 1712 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 0.82-1.02 (1H, m), 1.01-1.33 (4H, m), 1.35 (3H, s), 1.36-1.49 (1H, m), 1.50-1.60 (1H, m), 1.61-1.79 (4H, m), 1.94-2.15 (2H, m), 2.19 (3H, s), 2.28-2.36 (1H, m), 2.42-2.65 (4H, m); ¹³C NMR (100 MHz, CDCl₃) δ 23.1, 26.2, 26.5, 27.0, 28.5, 29.2, 30.2, 33.4, 33.8, 38.3, 40.4, 46.8, 89.1, 176.2, 207.7; MS (ESI-TOF) *m/z* 253 [M+H]⁺; HRMS calcd for C₁₅H₂₅O₃ [M+H]⁺, 253.1804; found, 253.1800.

5. Gas-phase acidity measurement

The gas-phase acidity measurements were performed on an Extrel FTMS 2001 Fourier transform mass spectrometer. Most of the experimental techniques used for the measurements of the equilibrium constants of the reversible proton-transfer reactions are the same as the procedures reported previously.¹⁸ Methyl nitrite as a chemical ionization reagent was not necessary for such strong acids. Electron impact of ionization produced the corresponding anions. The following schemes describe the sequence of reactions which occur in a typical experiment where AH and A₀H are the measured acid and the reference acid, respectively.



An experiment is initiated by a 5 ms pulse of a low-energy electron beam (0.3~0.5 eV) through the ICR cell. The partial pressures of the acids were maintained at lower than 4×10^{-7} Torr. The proton-transfer equilibrium (eq. 3) was achieved within 20~100 s of initiation of the reaction (depending on the pressure of neutrals). The equilibrium constant and free energy change for the reaction were evaluated by using the expression (eq. 4).

$$K = \frac{I(A^-) p(A_0H)}{I(A_0^-) p(AH)}$$
$$\Delta G_A = -RT \ln K \quad (4)$$

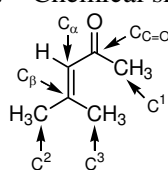
The relative abundances of ions A⁻ and A₀⁻ were determined by the relative intensities of ICR mass spectra signals when the equilibrium attained. The pressures of the neutral reactants were measured by means of a Bayard-Alpert type ionization gauge applying appropriate correction factors to correct the gauge reading for the different ionization cross sections of various compounds.¹⁹ The blank pressure was kept at less than 10^{-9} Torr. Each experiment was performed at several ratios of the partial pressures and at different overall pressures. The proton-transfer reactions were examined by ion-eject experiments. Equilibrium constants measured in this way can be used to calculate ΔG_{acid} at 300 K (eq. 4). The average uncertainty is ± 0.2 kcal/mol. The gas-phase acidity values for the reference compounds were taken from the literature.^{20,21} Gas-phase acidity value (295.7 kcal/mol) of Tf₂CHCH₂CHTf₂ was determined based on following free energy changes; 3 kcal/mol stronger than (F₃C)₅C₆OH (299.0),²⁰ 2.0 stronger than *m*-FC₆H₄CHTf₂ (298.1),²¹ 0.6 stronger than *m*-CF₃C₆H₄CHTf₂ (296.4),²¹ 0.2 stronger than *p*-CF₃C₆H₄CHTf₂ (295.9),²¹ 0.8 weaker than 3,5-(CF₃)₂C₆H₃CHTf₂ (294.9),²¹ where values in the parentheses are gas-phase acidities of the indicated reference acids.

6. NMR studies for mixtures of 2a and Brønsted acids

¹H and ¹³C NMR spectra of mixtures of **2a** (0.375 mmol) and Brønsted acid (0.375 mmol) in CDCl₃ (0.75 mL) were taken on a 400 MHz spectrometer at room temperature, and chemical shifts were reported in parts per million (ppm) using CHCl₃ (7.26 ppm) in CDCl₃ for ¹H NMR, and CDCl₃ (77.01 ppm) for ¹³C NMR as an internal

standard, respectively. Results are summarized in Tables S1 and S2.

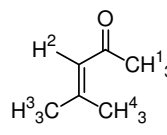
Table S1. Chemical shifts of **2a** in the presence of Brønsted acids in ^{13}C NMR (100 MHz)



Brønsted acid	Chemical shifts (ppm)											
	C _{C=O}	ΔC _{C=O}	C _α	ΔC _α	C _β	ΔC _β	C ¹	ΔC ¹	C ²	ΔC ²	C ³	ΔC ³
none	198.7	-	124.3	-	155.0	-	31.7	-	27.6	-	20.6	-
TfOH	210.6	+11.9	122.8	-1.5	186.8	+31.8	31.0	-0.7	28.3	+0.7	25.3	+4.7
Tf ₂ CHCH ₂ CHTf ₂	199.3	+0.6	124.2	-0.1	155.5	+0.5	31.5	-0.2	27.5	-0.1	20.5	-0.1
Tf ₂ CH ₂	199.0	+0.3	124.2	-0.1	155.5	+0.5	31.5	-0.2	27.5	-0.1	20.5	-0.1

Conditions: **2a** (0.375 mmol) and Brønsted acids (0.375 mmol) in CDCl₃ (0.75 mL) at room temperature.

Table S2. Chemical shifts of **2a** in the presence of Brønsted acids in ^1H NMR (400 MHz)

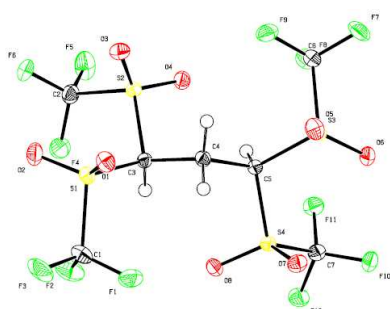


Brønsted acid	Chemical shifts (ppm)							
	H ¹	ΔH ¹	H ²	ΔH ²	H ³	ΔH ³	H ⁴	ΔH ⁴
none	2.15	-	6.08	-	2.13	-	1.87	-
TfOH	2.69	+0.54	6.56	+0.48	2.45	+0.32	2.29	+0.42
Tf ₂ CH ₂	2.12	-0.03	6.06	-0.02	2.09	-0.04	1.85	-0.02
Tf ₂ CHCH ₂ CHTf ₂	2.12	-0.03	6.06	-0.02	2.08	-0.05	1.85	-0.02

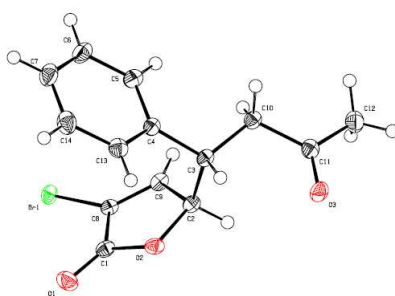
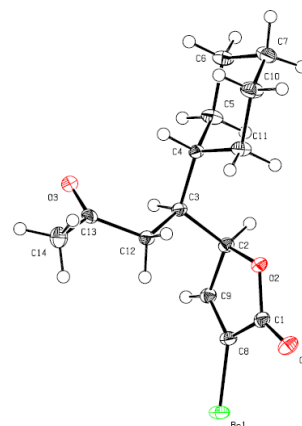
Conditions: **2a** (0.375 mmol) and Brønsted acids (0.375 mmol) in CDCl₃ (0.75 mL) at room temperature.

7. X-ray crystallographic data of 1, *anti*-3fe, *anti*-3ie, *anti*-3je, *anti,anti*-3oe, and *anti*-3if

Crystallographic data for the X-ray crystal structure analysis of **1**, ***anti-3fe***, ***anti-3ie***, ***anti-3je***, ***anti,anti-3oe***, and ***anti-3if*** have been deposited with Cambridge Crystallographic Data Center (CCDC) as supplementary publication Nos. CCDC 674990 (**1**), 739547 (***anti-3fe***), 739352 (***anti-3ie***), 739353 (***anti-3je***), 739351 (***anti,anti-3oe***) and 739354 (***anti-3if***). These data can be obtained free of charge from the CCDC *via* www.ccdc.cam.ac.uk/data_request/cif.



X-ray structure of **1**

X-ray structure of *anti*-3fe

X-ray structure of *anti*-3ie

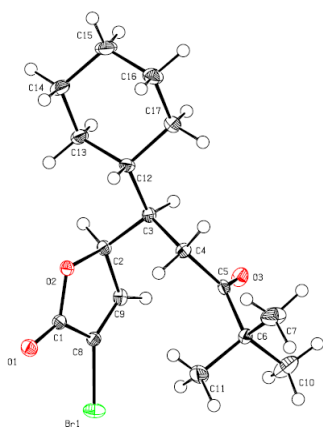
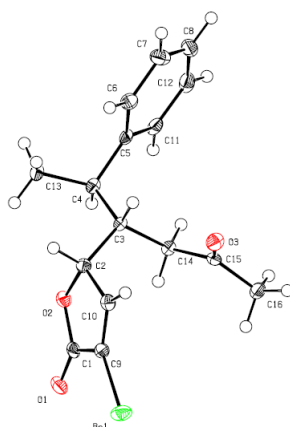
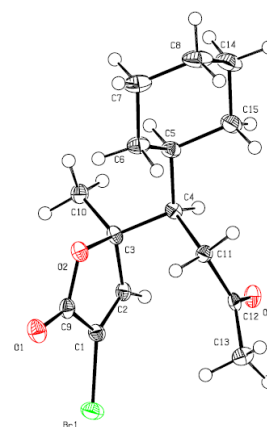
X-ray structure of *anti*-3jeX-ray structure of *anti,anti*-3oeX-ray structure of *anti*-**3if**

Table S3. Crystal data and structure refinement for **1**.

Empirical formula	$\text{C}_7\text{H}_4\text{F}_{12}\text{O}_8\text{S}_4$		
Formula weight	572.34		
Temperature	100 K		
Wavelength	0.71073 Å		
Crystal system	Monoclinic		
Space group	P2(1)/c		
Unit cell dimensions	a = 9.9367(9) Å		$\alpha = 90^\circ$

	$b = 16.5820(15) \text{ \AA}$	$\beta = 103.5850(10)^\circ$
	$c = 10.9892(10) \text{ \AA}$	$\gamma = 90^\circ$
Volume	$1760.0(3) \text{ \AA}^3$	
Z	4	
Density (calculated)	2.160 Mg/m^3	
Absorption coefficient	0.702 mm^{-1}	
F(000)	1128	
Crystal size	$0.22 \times 0.16 \times 0.05 \text{ mm}^3$	
Theta range for data collection	2.11 to 27.48°	
Index ranges	$-12 \leq h \leq 10$, $-21 \leq k \leq 20$, $-9 \leq l \leq 14$	
Reflections collected	9655	
Independent reflections	3932 [R(int) = 0.0237]	
Completeness to theta = 27.48°	97.4 %	
Absorption correction	Empirical	
Max. and min. transmission	0.9657 and 0.8609	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3932 / 0 / 280	
Goodness-of-fit on F^2	1.021	
Final R indices [I > 2sigma(I)]	R1 = 0.0284, wR2 = 0.0621	
R indices (all data)	R1 = 0.0384, wR2 = 0.0666	
Largest diff. peak and hole	0.421 and $-0.400 \text{ e.\AA}^{-3}$	

Table S4. Crystal data and structure refinement for *anti-3fe*.

Empirical formula	$\text{C}_{14}\text{H}_{13}\text{BrO}_3$	
Formula weight	309.15	
Temperature	90 K	
Wavelength	0.71073 \AA	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	$a = 6.2196(16) \text{ \AA}$	$\alpha = 73.247(2)^\circ$
	$b = 10.038(3) \text{ \AA}$	$\beta = 85.069(3)^\circ$
	$c = 10.899(3) \text{ \AA}$	$\gamma = 89.251(3)^\circ$
Volume	$649.1(3) \text{ \AA}^3$	
Z	2	
Density (calculated)	1.582 Mg/m^3	
Absorption coefficient	3.164 mm^{-1}	
F(000)	312	
Crystal size	$0.23 \times 0.08 \times 0.03 \text{ mm}^3$	

Theta range for data collection	1.96 to 27.69°.
Index ranges	-8<=h<=8, -12<=k<=12, -13<=l<=14
Reflections collected	6933
Independent reflections	2937 [R(int) = 0.0521]
Completeness to theta = 27.69°	96.8 %
Absorption correction	Empirical
Max. and min. transmission	0.9111 and 0.5298
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	2937 / 0 / 164
Goodness-of-fit on F ²	1.104
Final R indices [I>2sigma(I)]	R1 = 0.0370, wR2 = 0.1106
R indices (all data)	R1 = 0.0383, wR2 = 0.1117
Largest diff. peak and hole	0.862 and -1.490 e.Å ⁻³

Table S5. Crystal data and structure refinement for compound *anti-3ie*.

Empirical formula	C ₁₄ H ₁₉ BrO ₃	
Formula weight	315.20	
Temperature	90 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21/n	
Unit cell dimensions	a = 6.2821(12) Å	α = 90°.
	b = 15.603(3) Å	β = 101.736(2)°.
	c = 14.722(3) Å	γ = 90°.
Volume	1412.9(5) Å ³	
Z	4	
Density (calculated)	1.482 Mg/m ³	
Absorption coefficient	2.908 mm ⁻¹	
F(000)	648	
Crystal size	0.49 x 0.37 x 0.33 mm ³	
Theta range for data collection	1.92 to 27.51°.	
Index ranges	-8<=h<=8, -20<=k<=20, -18<=l<=10	
Reflections collected	7760	
Independent reflections	3188 [R(int) = 0.0285]	
Completeness to theta = 27.51°	98.3 %	
Absorption correction	Empirical	
Max. and min. transmission	0.4471 and 0.3300	
Refinement method	Full-matrix least-squares on F ²	

Data / restraints / parameters	3188 / 0 / 164
Goodness-of-fit on F^2	1.051
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0270$, $wR2 = 0.0707$
R indices (all data)	$R1 = 0.0318$, $wR2 = 0.0729$
Largest diff. peak and hole	0.783 and -0.583 e.Å ⁻³

Table S6. Crystal data and structure refinement for compound *anti-3je*.

Empirical formula	$C_{17}H_{25}BrO_3$	
Formula weight	357.28	
Temperature	90 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21/c	
Unit cell dimensions	$a = 9.477(3)$ Å	$\alpha = 90^\circ$.
	$b = 10.226(3)$ Å	$\beta = 93.780(3)^\circ$.
	$c = 17.617(5)$ Å	$\gamma = 90^\circ$.
Volume	$1703.6(9)$ Å ³	
Z	4	
Density (calculated)	1.393 Mg/m ³	
Absorption coefficient	2.420 mm ⁻¹	
F(000)	744	
Crystal size	0.23 x 0.23 x 0.04 mm ³	
Theta range for data collection	2.15 to 27.69°.	
Index ranges	-12 ≤ h ≤ 12, -6 ≤ k ≤ 13, -22 ≤ l ≤ 21	
Reflections collected	9346	
Independent reflections	3894 [$R(\text{int}) = 0.0542$]	
Completeness to $\theta = 27.69^\circ$	97.4 %	
Absorption correction	Numerical	
Max. and min. transmission	0.9094 and 0.6060	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3894 / 0 / 193	
Goodness-of-fit on F^2	1.021	
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0362$, $wR2 = 0.0964$	
R indices (all data)	$R1 = 0.0470$, $wR2 = 0.1023$	
Largest diff. peak and hole	0.545 and -0.398 e.Å ⁻³	

Table S7. Crystal data and structure refinement for compound *anti,anti-3oe*.

Empirical formula	$C_{16}H_{17}BrO_3$	
Formula weight	337.21	
Temperature	90 K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P b c n	
Unit cell dimensions	a = 21.609(4) Å	$\alpha = 90^\circ$.
	b = 8.9577(16) Å	$\beta = 90^\circ$.
	c = 15.245(3) Å	$\gamma = 90^\circ$.
Volume	2951.0(9) Å ³	
Z	8	
Density (calculated)	1.518 Mg/m ³	
Absorption coefficient	2.790 mm ⁻¹	
F(000)	1376	
Crystal size	0.23 x 0.19 x 0.16 mm ³	
Theta range for data collection	1.88 to 27.58°.	
Index ranges	-28 ≤ h ≤ 18, -11 ≤ k ≤ 11, -15 ≤ l ≤ 19	
Reflections collected	15617	
Independent reflections	3397 [R(int) = 0.0554]	
Completeness to theta = 27.58°	99.5 %	
Absorption correction	Numerical	
Max. and min. transmission	0.6638 and 0.5662	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	3397 / 0 / 183	
Goodness-of-fit on F ²	1.027	
Final R indices [I > 2σ(I)]	R1 = 0.0303, wR2 = 0.0759	
R indices (all data)	R1 = 0.0398, wR2 = 0.0811	
Largest diff. peak and hole	0.634 and -0.491 e.Å ⁻³	

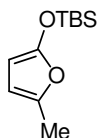
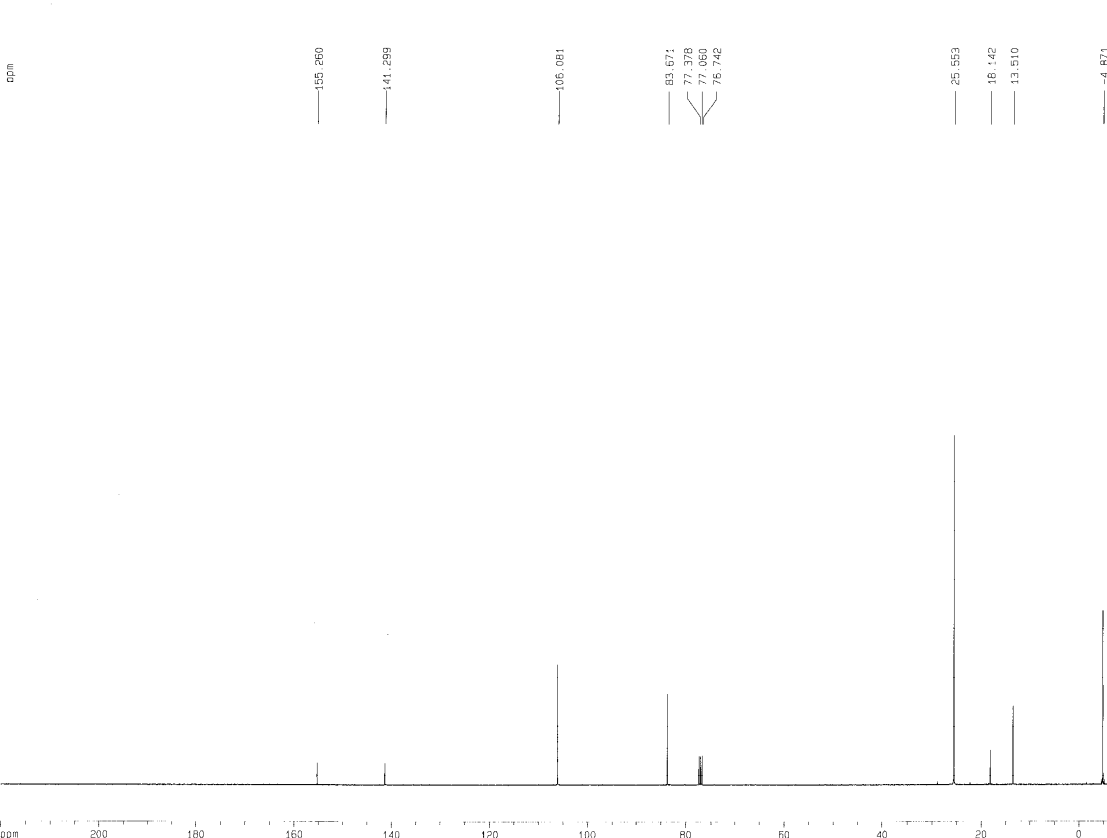
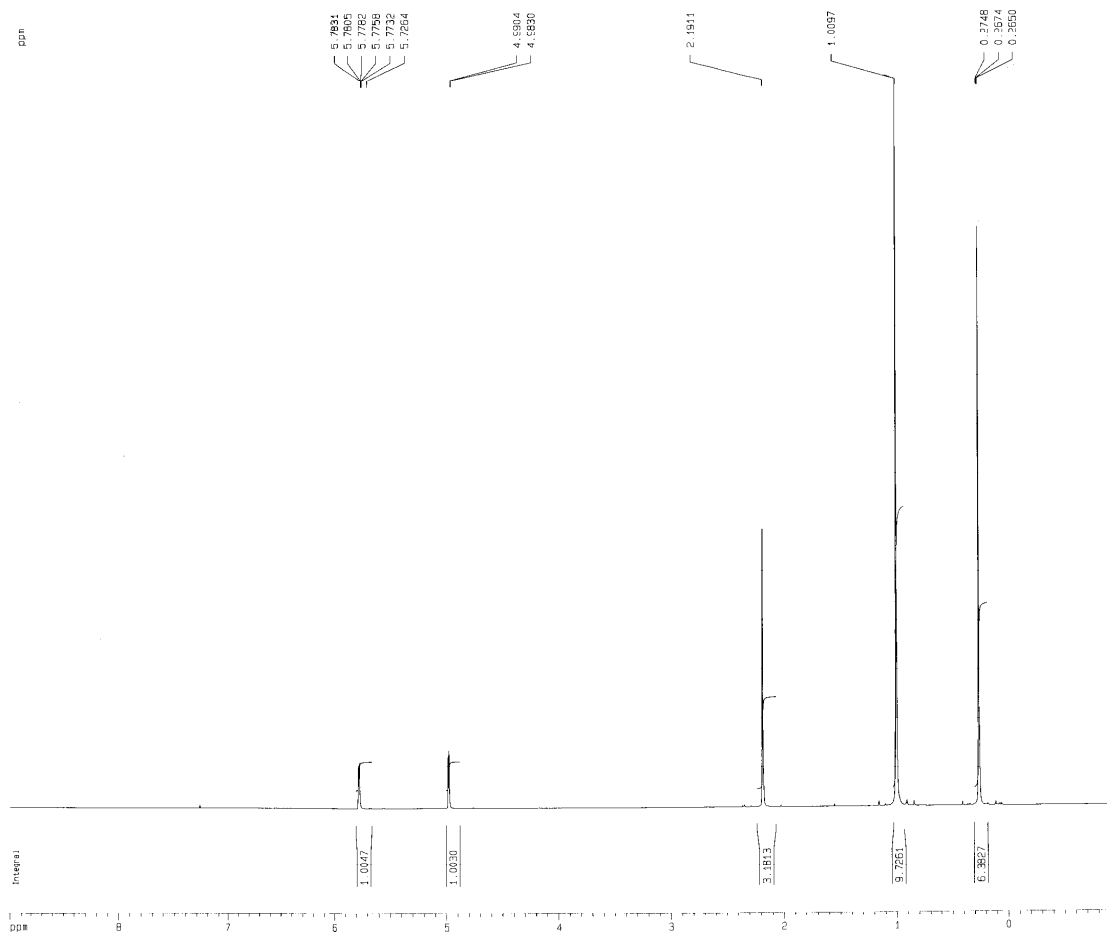
Table S8. Crystal data and structure refinement for compound *anti-3if*.

Empirical formula	$C_{15}H_{21}BrO_3$	
Formula weight	329.23	
Temperature	90 K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P c a 21	
Unit cell dimensions	a = 20.354(6) Å	$\alpha = 90^\circ$.
	b = 10.517(3) Å	$\beta = 90^\circ$.

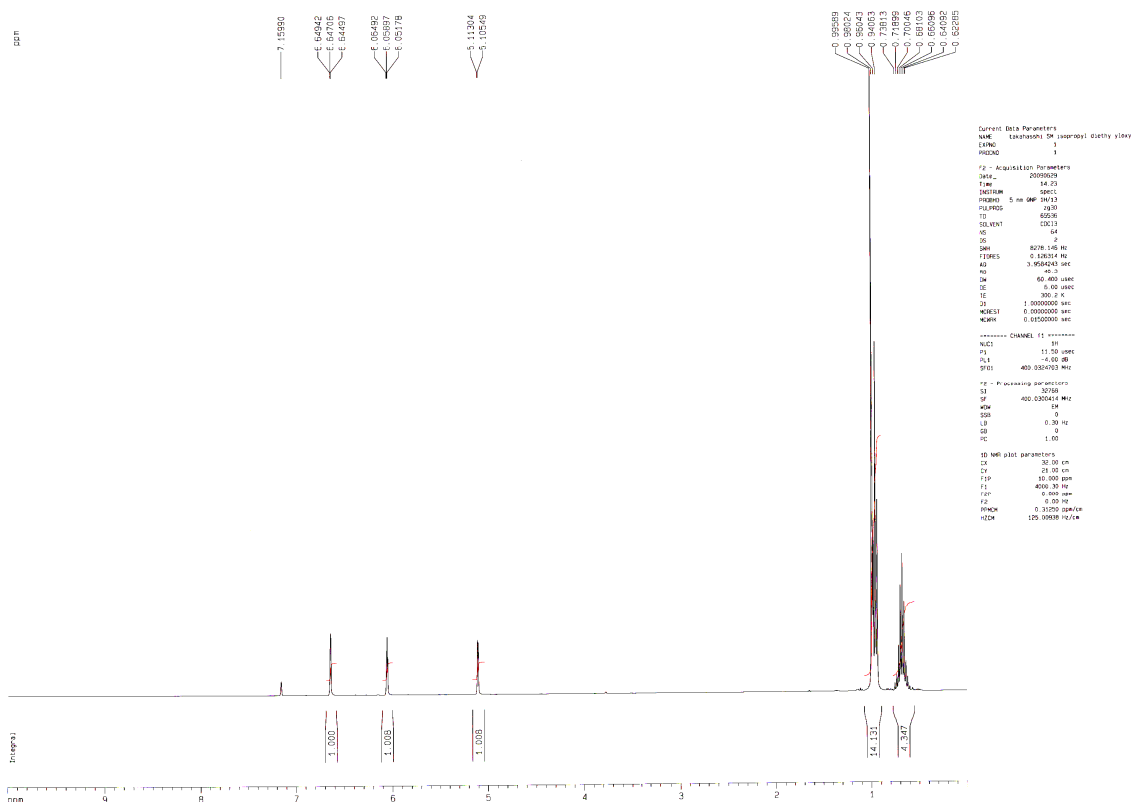
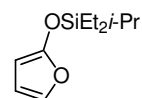
	$c = 7.131(2) \text{ \AA}$	$\gamma = 90^\circ$.
Volume	$1526.3(8) \text{ \AA}^3$	
Z	4	
Density (calculated)	1.433 Mg/m^3	
Absorption coefficient	2.695 mm^{-1}	
F(000)	680	
Crystal size	$0.28 \times 0.20 \times 0.05 \text{ mm}^3$	
Theta range for data collection	1.94 to 27.81° .	
Index ranges	$-26 \leq h \leq 26$, $-13 \leq k \leq 13$, $-9 \leq l \leq 9$	
Reflections collected	16690	
Independent reflections	3579 [R(int) = 0.0386]	
Completeness to $\theta = 27.81^\circ$	99.4 %	
Absorption correction	Numerical	
Max. and min. transmission	0.8770 and 0.5191	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3579 / 1 / 175	
Goodness-of-fit on F^2	1.036	
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0259$, $wR2 = 0.0625$	
R indices (all data)	$R1 = 0.0306$, $wR2 = 0.0646$	
Absolute structure parameter	0.364(7)	
Largest diff. peak and hole	0.698 and $-0.318 \text{ e.\AA}^{-3}$	

45 64151 1111111111

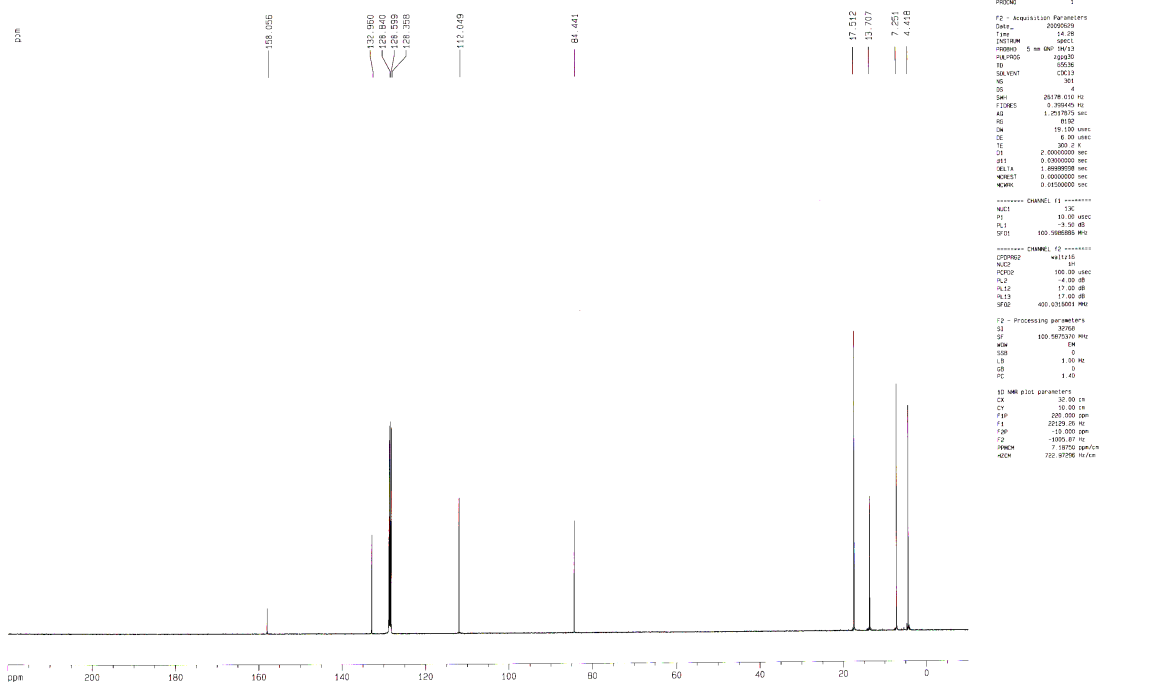




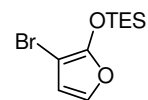
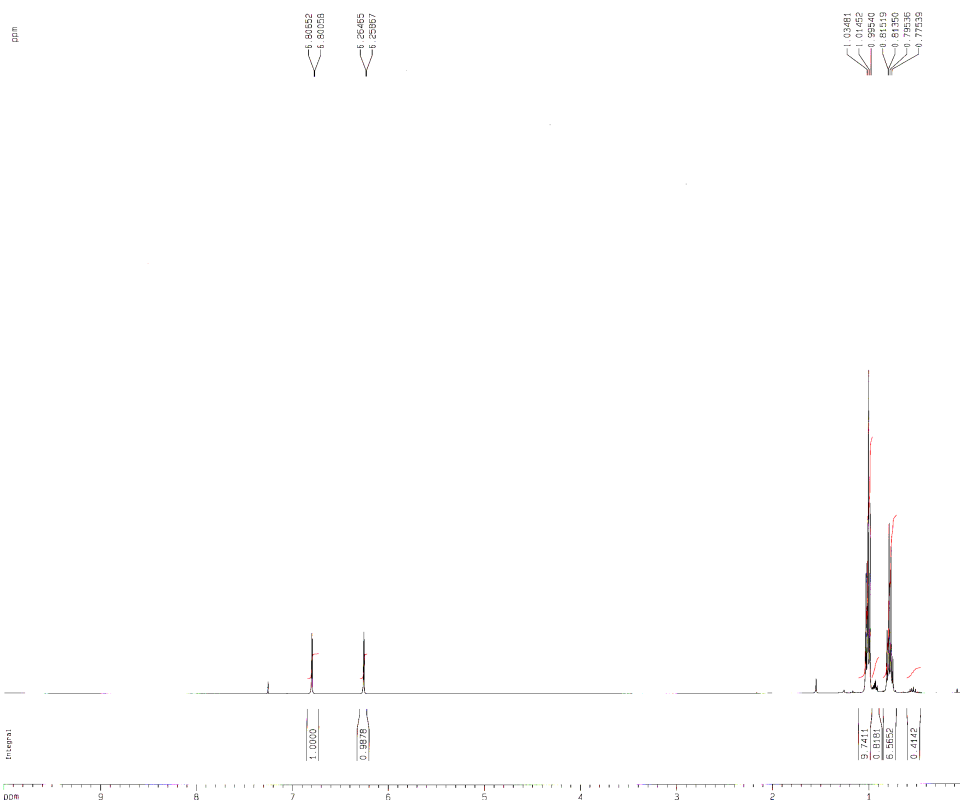
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DPX400 carbon



JF4000 proton



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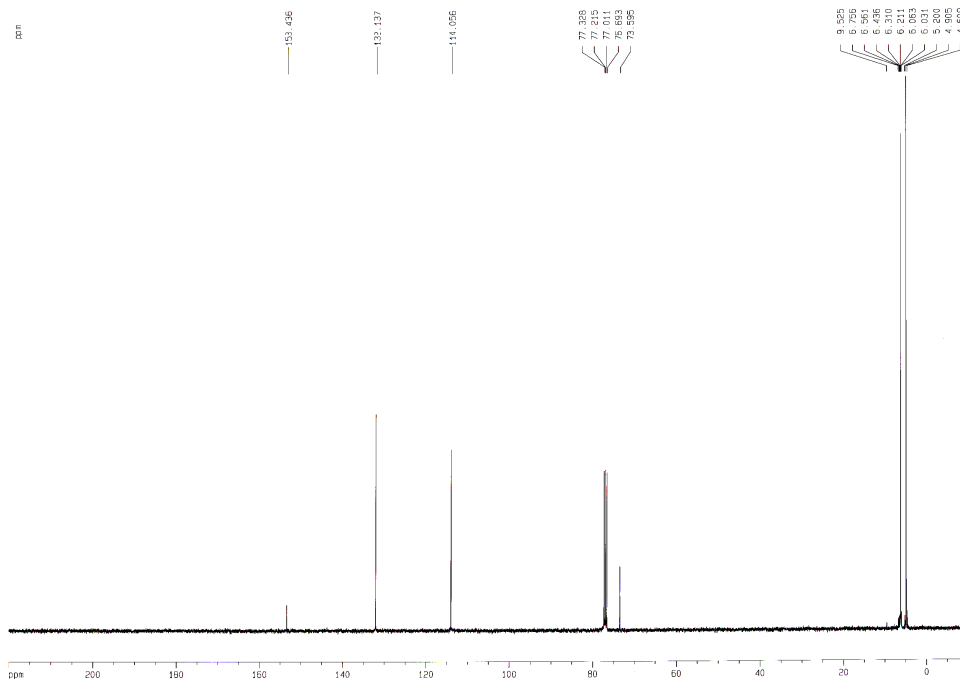
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DPX400 carbon



Current Data Parameters
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PROCNO 1

F2 - Acquisition Parameters
Date_ 20050606
Time 14.22
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 26378.010 Hz
FIDRES 0.399445 Hz
AQ 1.251970 sec
RG 819.4
DE 5.00 usec
TE 300.2 K
DT 2.0000000 sec
MCREST 0.0300000 sec
DELTA 1.9999999 sec
MCWEN 0.0000000 sec
MCWEN 0.0150000 sec

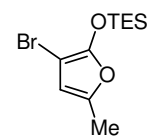
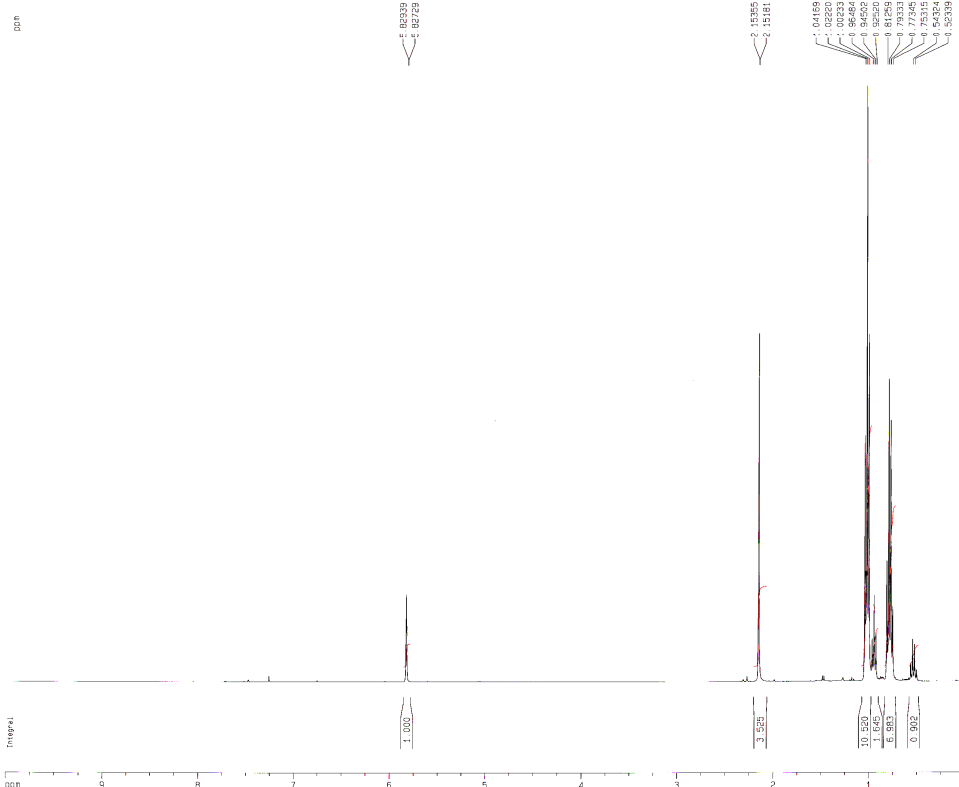
----- CHANNEL f1 -----
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.585898 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.015000 MHz

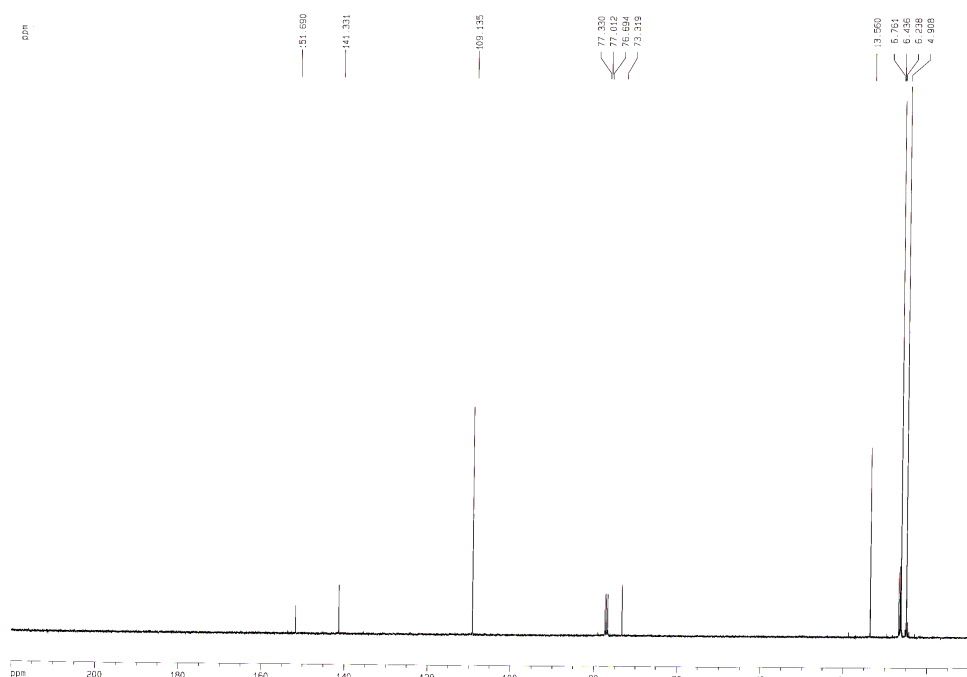
F2 - Processing parameters
SI 32768
SF 100.5876230 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 38.00 cm
CY 18.00 cm
F1P 220.000 ppm
F1 201.2920 Hz
F2P -10.000 ppm
F2 -100.588 Hz
PRCM 7.18750 ppm/cm
HZCM 722.57357 Hz/cm

DPX400 proton

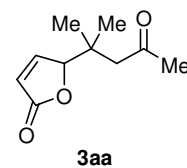
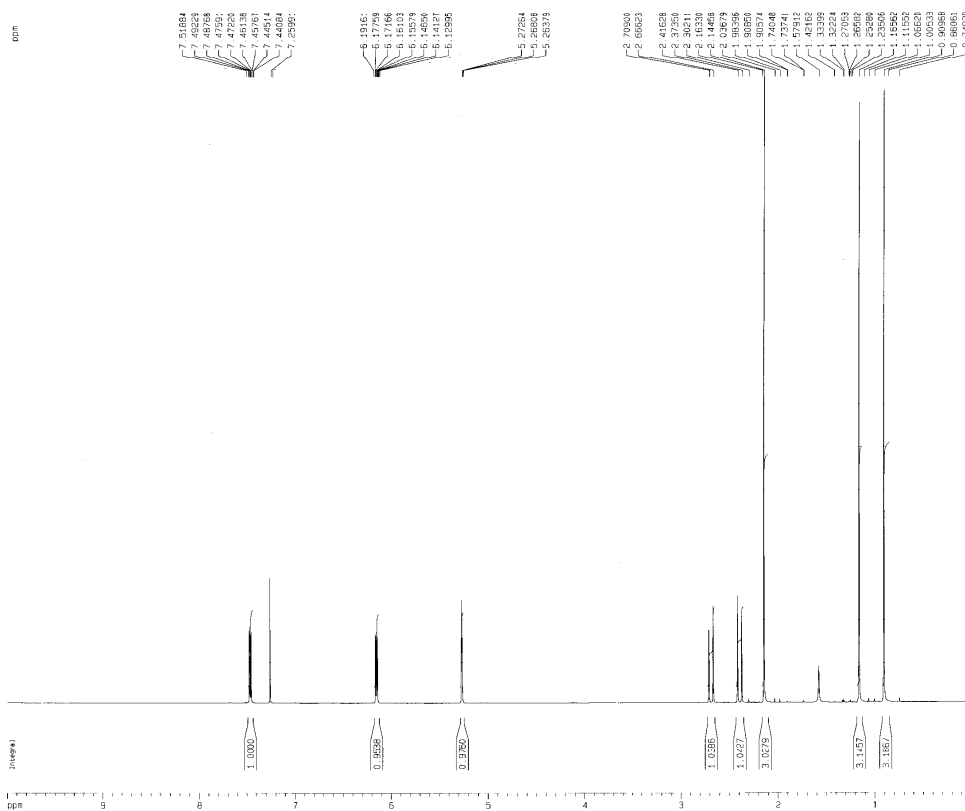


DPX400 carbon



9. ^1H and ^{13}C NMR spectra of VMM adducts

untreated product



Current Data Parameters
NAME takahashi001
EXPNO 1
PROCNO 1

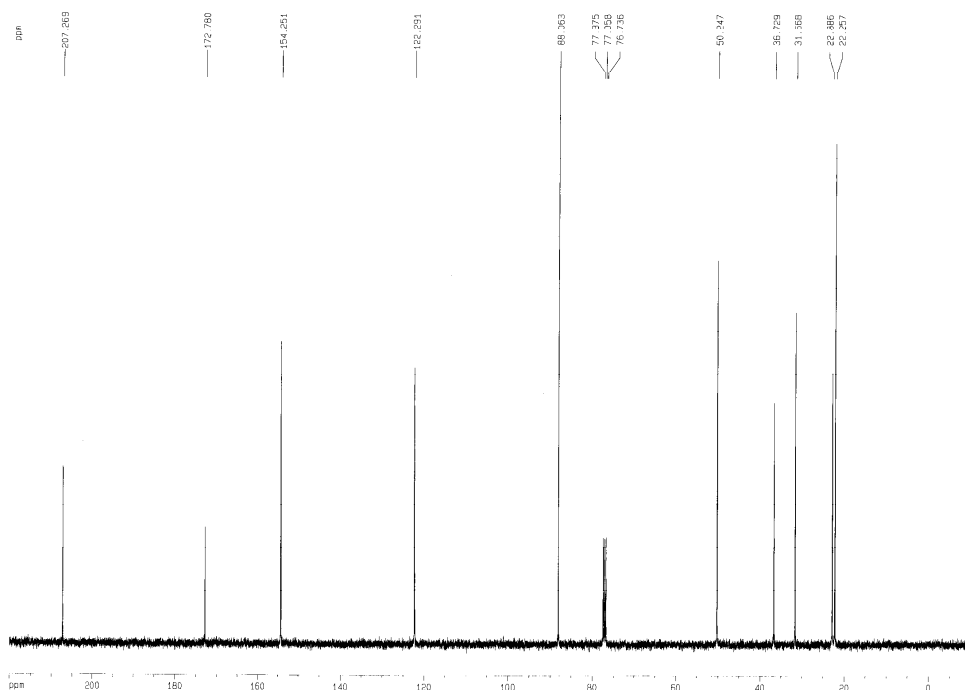
F2 - Acquisition Parameters
Date_ 20070822
Time 17.25
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 9270.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DM 50.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.0000000 sec
MCREST 0.0000000 sec
MCWK 0.0150000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.030067 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 21.00 cm
EXR 10.000 ppm
F1 4000.30 Hz
F2 0.000 ppm
F3 0.00 Hz
P1PCW 0.31250 ppm/cm
HZCM 125.06938 Hz/cm

DPX400 carbon



Current Data Parameters
NAME takahashi001
EXPNO 2
PROCNO 1

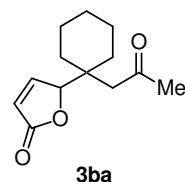
F2 - Acquisition Parameters
Date_ 20070822
Time 16.50
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 113
DS 1
SWH 25175.910 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 16384
DM 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTA 1.80999998 sec
MCREST 0.0000000 sec
MCWK 0.0150000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.6261800 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0150001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876371 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 27.00 cm
F1P 220.000 ppm
F1 22129.26 Hz
F2 -10.000 ppm
F3 -1005.88 Hz
F2 7.149760 mm/cm
HZCM 722.97363 Hz/cm



```

Current Data Parameters
NAME AGE009
EXPNO 1
PROCNO 1

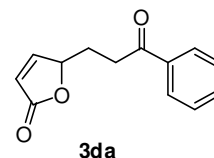
F2 - Acquisition Parameters
Date_ 20060110
Time 19:34
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
HWD 1000.00
TO SOLVENT CDCl3
NS 164
DS 4
SWH 21718.010 MHz
FIDRES 0.39445 Hz
AQ 1.2201694 sec
RG 364.5 I
DE 19.100 usec
TE 300.2 K
DW 2.000 usec
d1 2.0000000 sec
d11 0.0030000 sec
DELTA 1.8999999 sec
NUC1 13C
NUC2 1H
SFO1 100.5080600 MHz
===== CHANNEL f1 =====
P1 10.000 usec
PD1 0.0010001 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPDQ 2.000 usec
SF 100.5087600 MHz
PL1 17.00 dB
PL2 17.00 dB
PL3 17.00 dB
SFO2 400.0130001 MHz
===== CHANNEL f3 =====
CPDPRG2 waltz16
NUC2 1H
PCPDQ 2.000 usec
SF 100.5087600 MHz
PL1 17.00 dB
PL2 17.00 dB
PL3 17.00 dB
SFO2 400.0130001 MHz
===== CHANNEL f4 =====
CPDPRG2 waltz16
NUC2 1H
PCPDQ 2.000 usec
SF 100.5087600 MHz
PL1 17.00 dB
PL2 17.00 dB
PL3 17.00 dB
SFO2 400.0130001 MHz

F2 - Processing parameters
SF 376.768 MHz
SF2 100.5087600 MHz
WDW EM
SSB 0
GB 0
PC 1.40

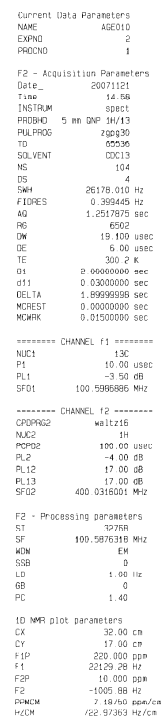
10 spin plot parameters
CX 32.00 cm
CY 17.00 cm
CZ 220.000 ppm
H1 220.193 Hz
F2P -100.000 ppm
F2 7.95658 Hz
H2P 7.95658 ppm
H2CM 7.95658 Hz/cm
H2CM 7.95658 Hz/cm

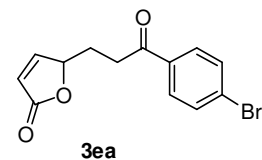
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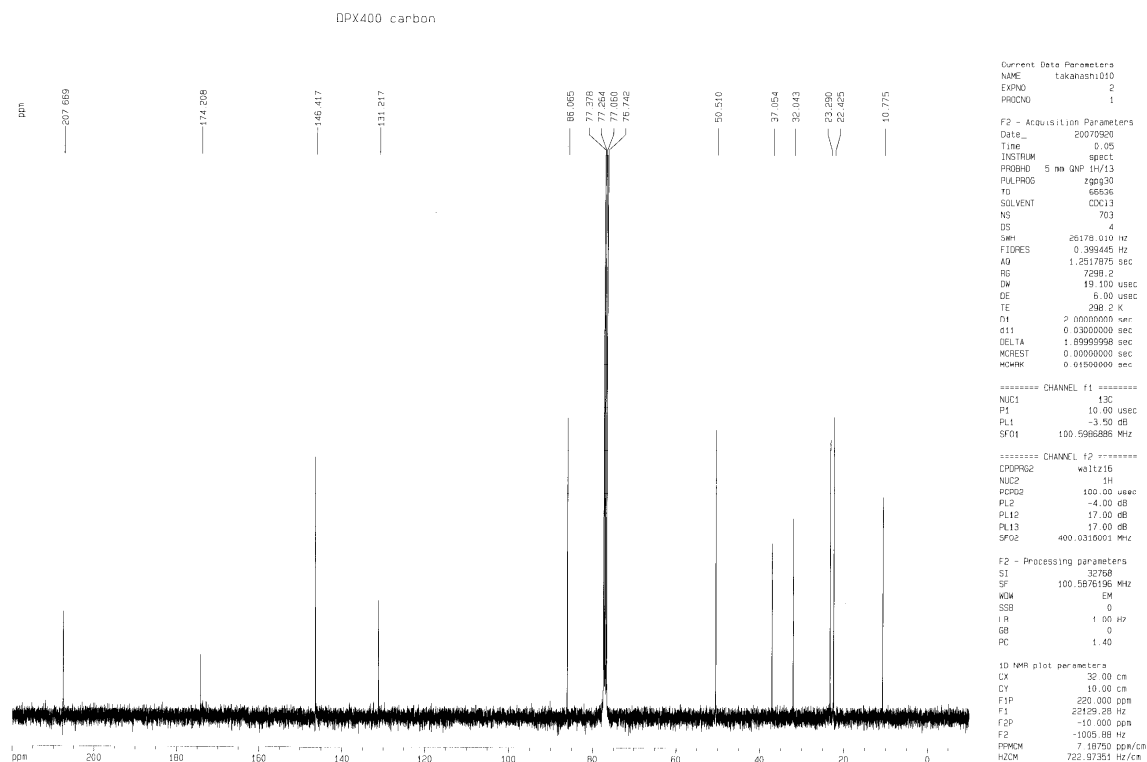
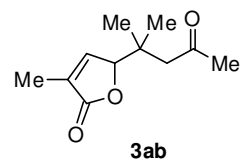
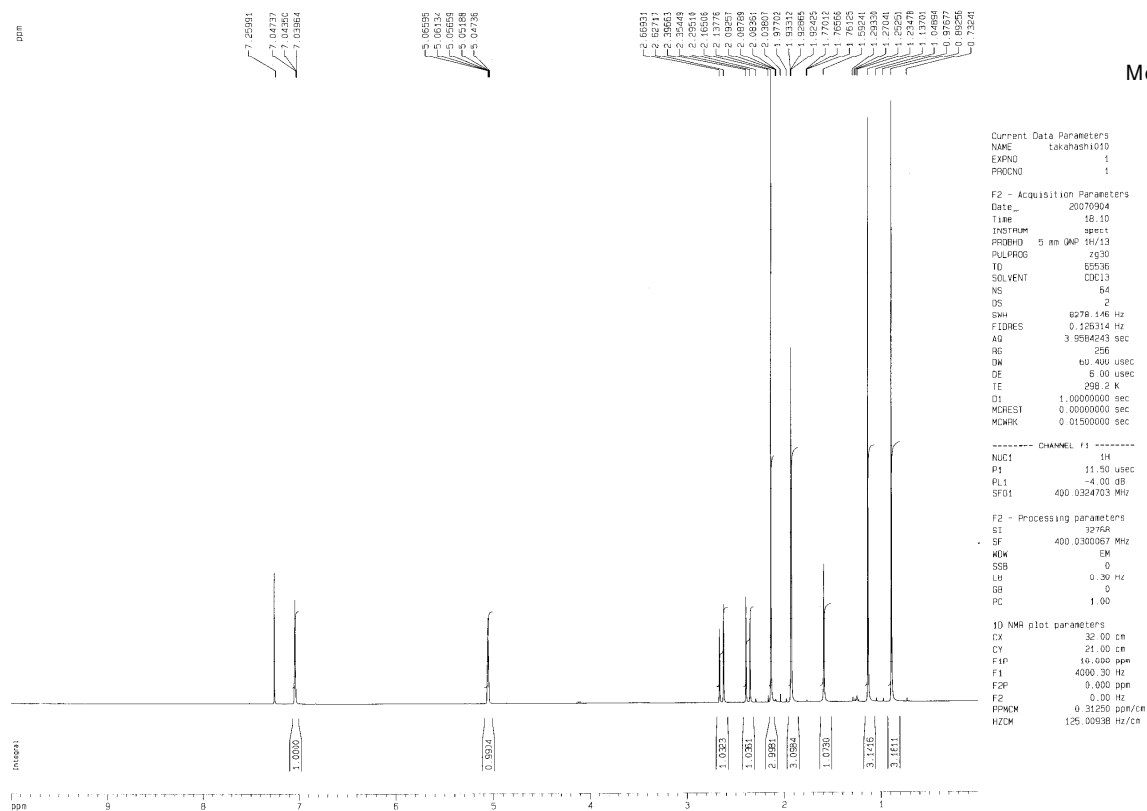
DPX400 carbon



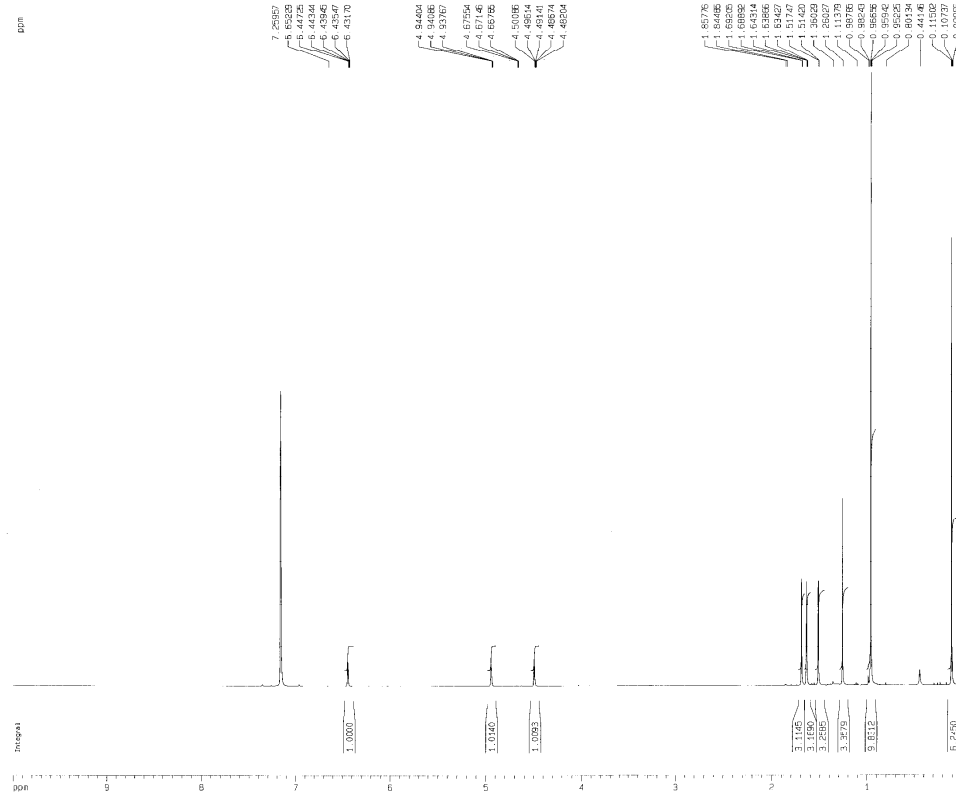


DPX400 carbon

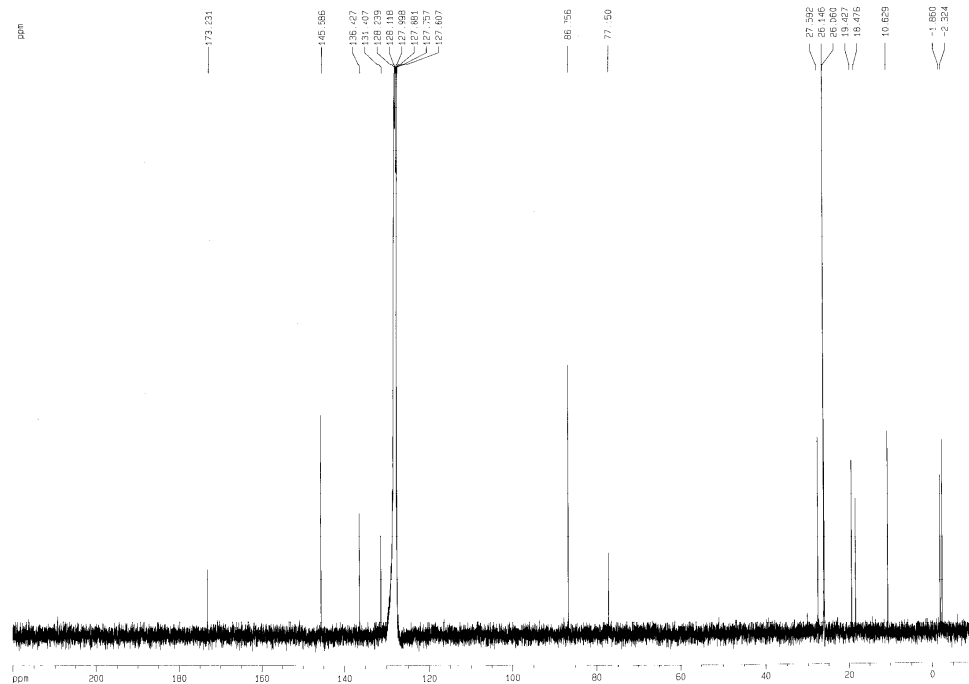




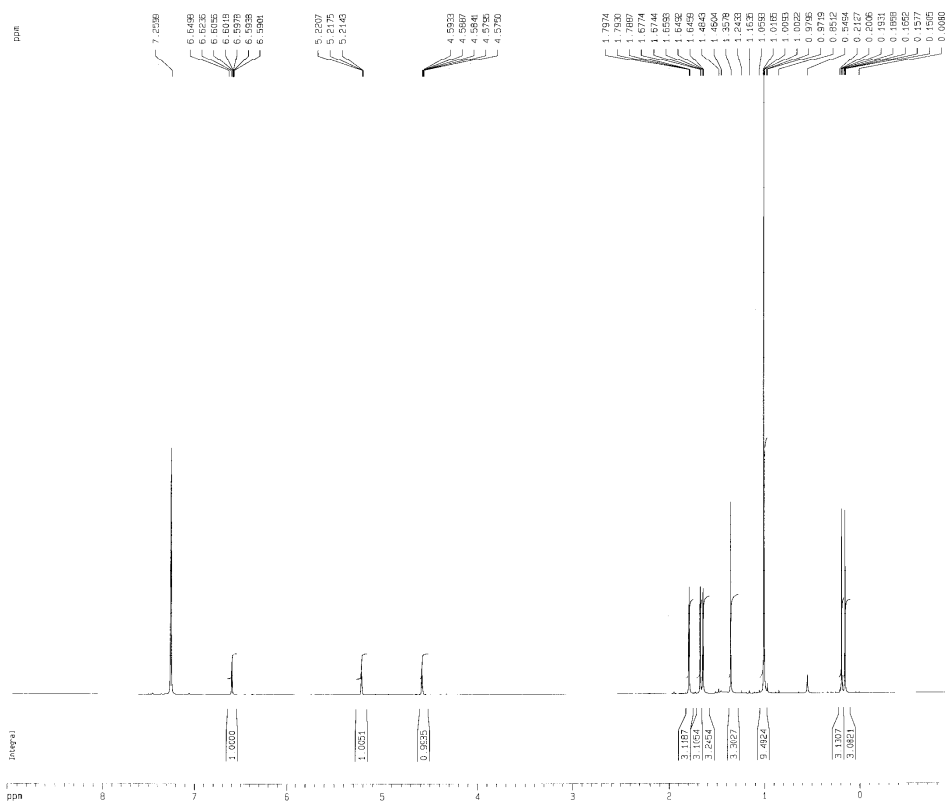
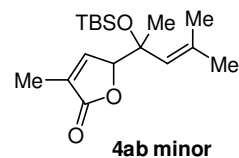
0.00000000



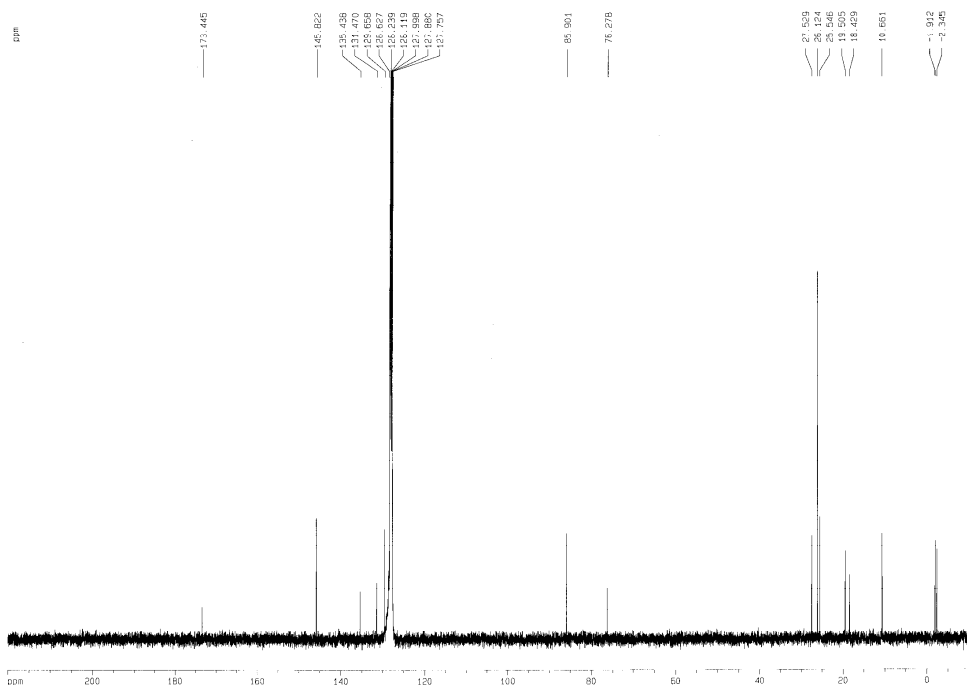
DPX400 carbon

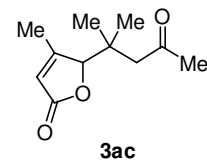
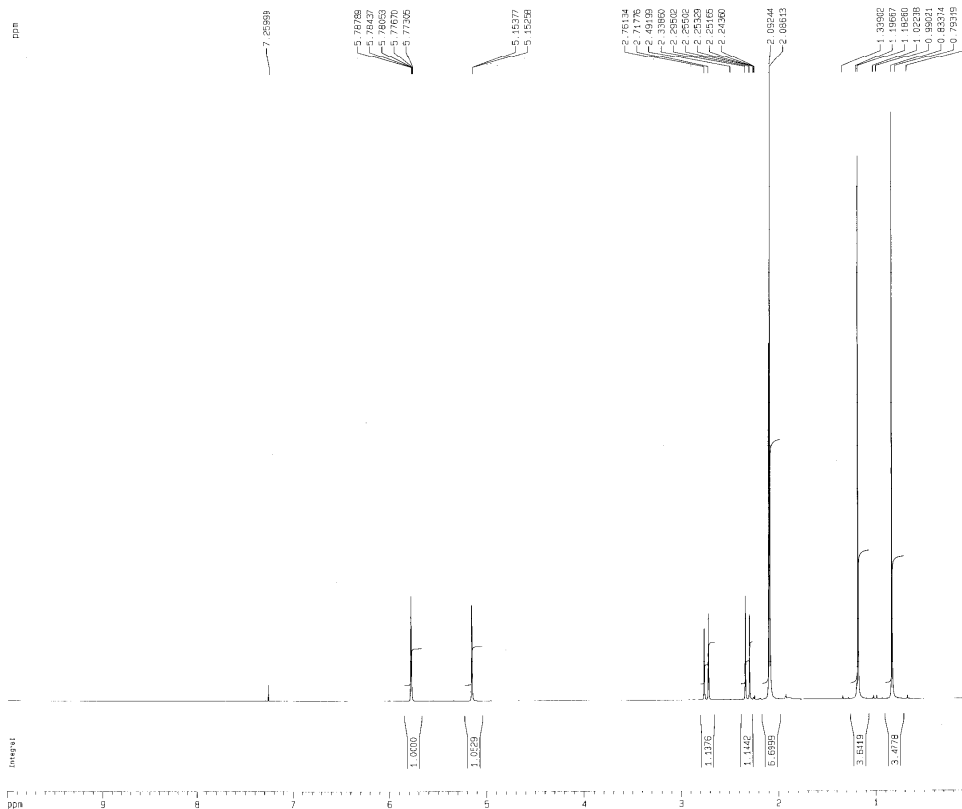


01/10/2007 11:00:00

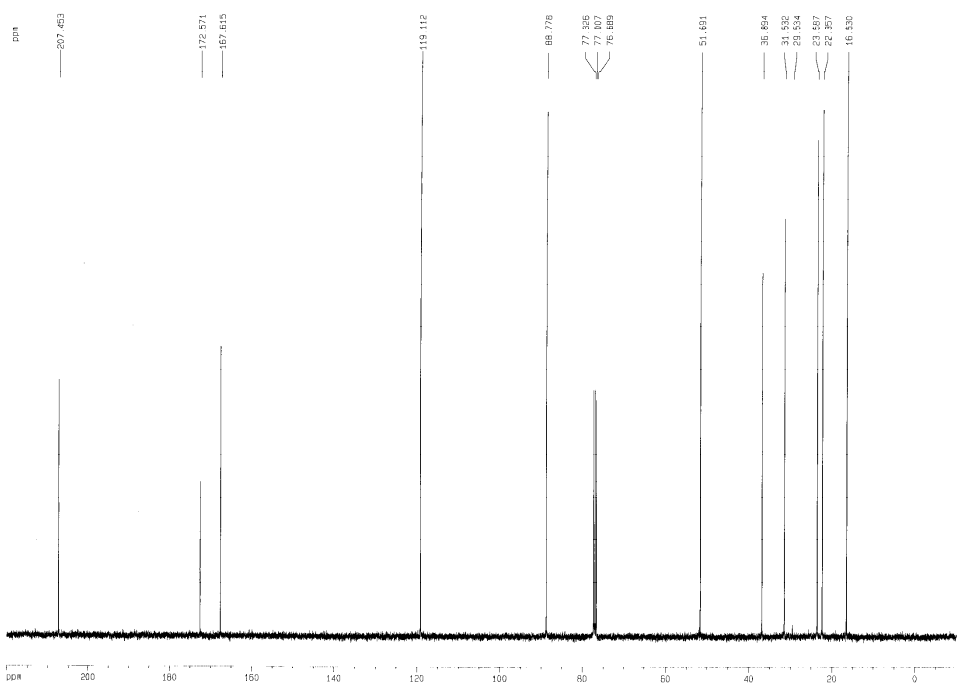


DPX400 carbon

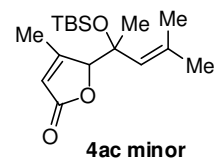
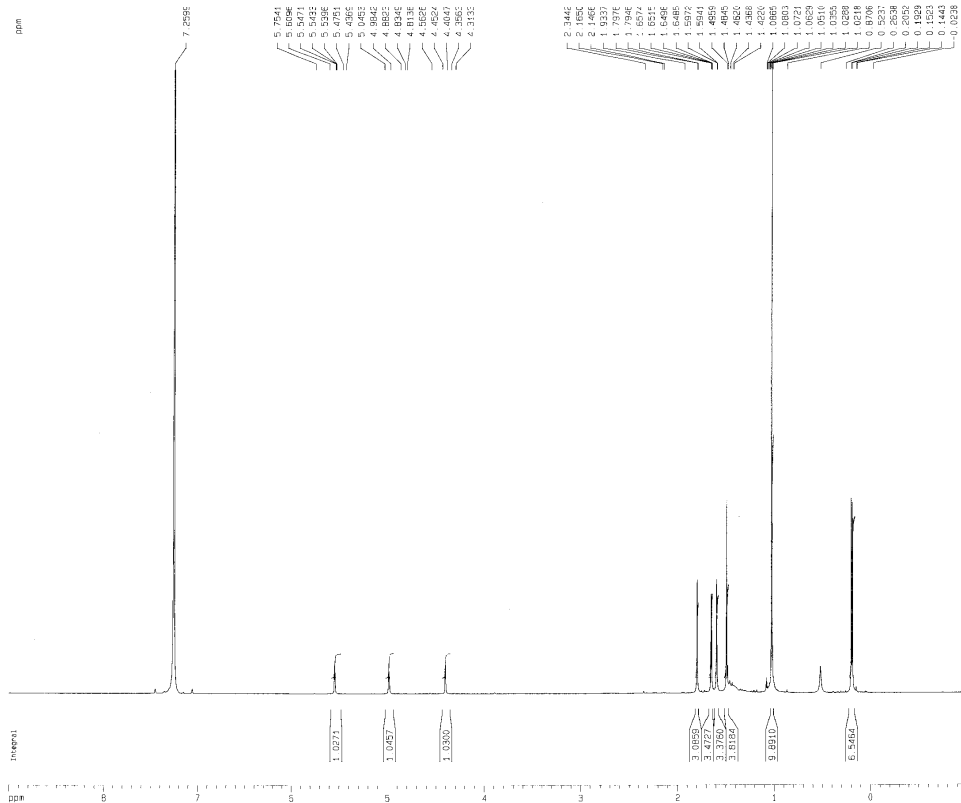




DPX400 carbon



5 7544



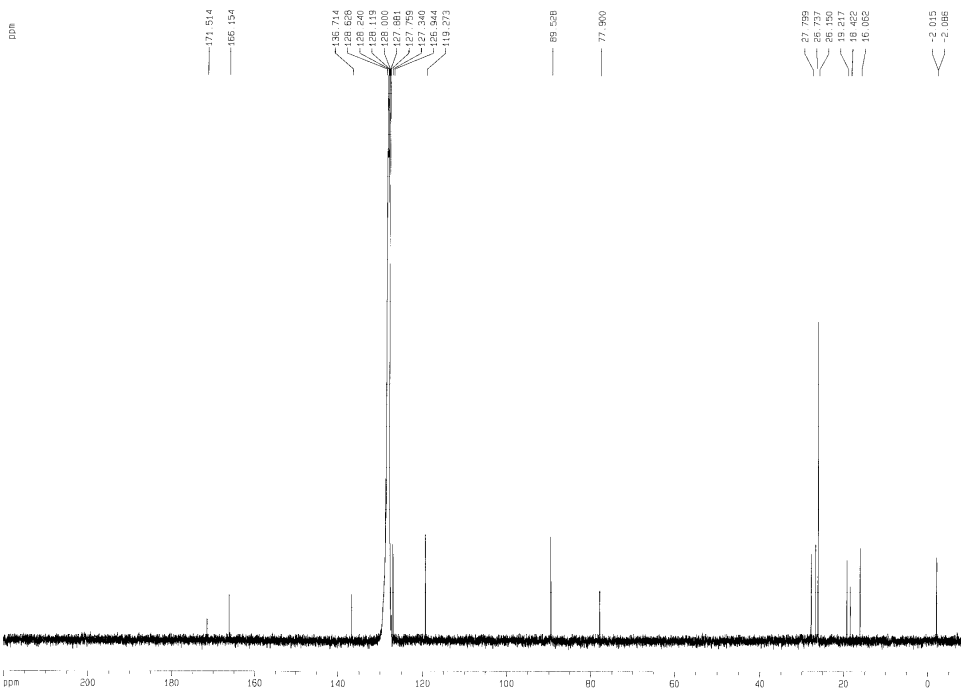
Current Data Parameters
NAME AGE045 minor
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20071205
Time 0.12
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 287.4
DM 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRR 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 11.50 usec
PL1 4.00 dB
SFO1 400.034703 MHz

F2 - Processing parameters
SI 32768
SF 400.030017 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 21.00 cm
F1P 9.000 ppm
F1 3600.27 Hz
F2P -1.000 ppm
F2 -400.03 Hz
PRCM 0.31250 ppm/cm
HZCM 125.00938 Hz/cm



Current Data Parameters
NAME AGE045 minor
EXPNO 1
PROCNO 1

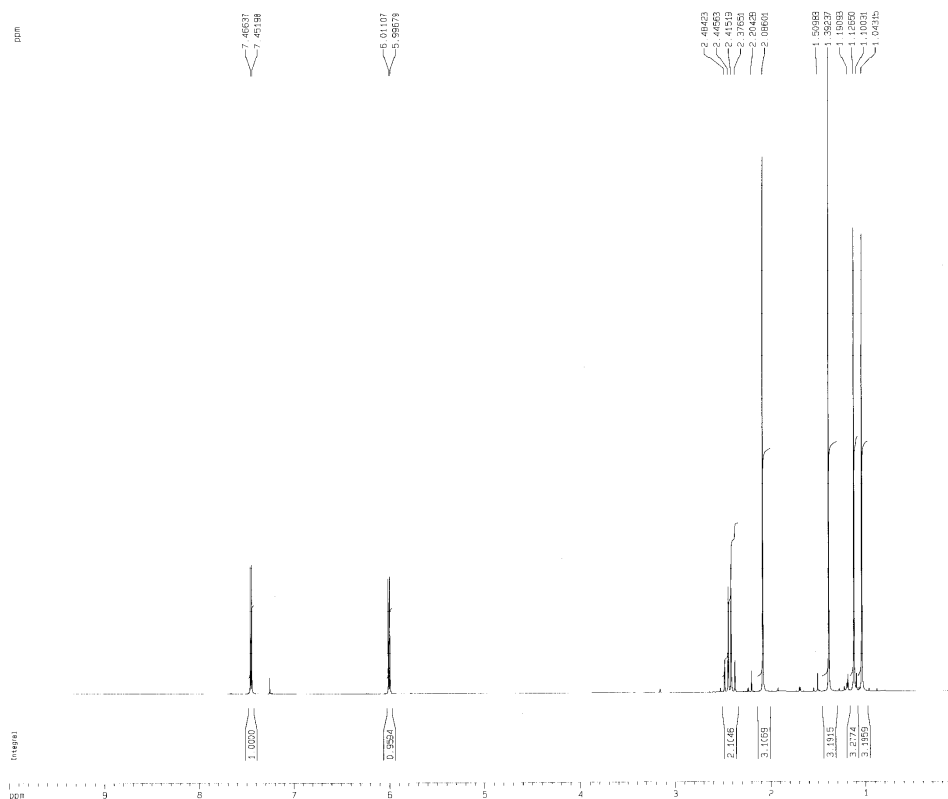
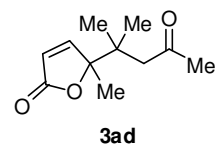
F2 - Acquisition Parameters
Date_ 20071205
Time 0.51
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
SWH 26178.010 Hz
FIDRES 0.394445 Hz
AQ 1.2517875 sec
RG 8192
DM 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.00000000 sec
MCREST 0.00000000 sec
MCWRR 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986896 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5976028 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 0.00 cm
F1P 220.000 ppm
F1 26169.27 Hz
F2P -10.000 ppm
F2 -100.58 Hz
PRCM 7.18750 ppm/cm
HZCM 722.97333 Hz/cm



Current Data Parameters
NAME takahashi009
EXPNO 1
PROCNO 1

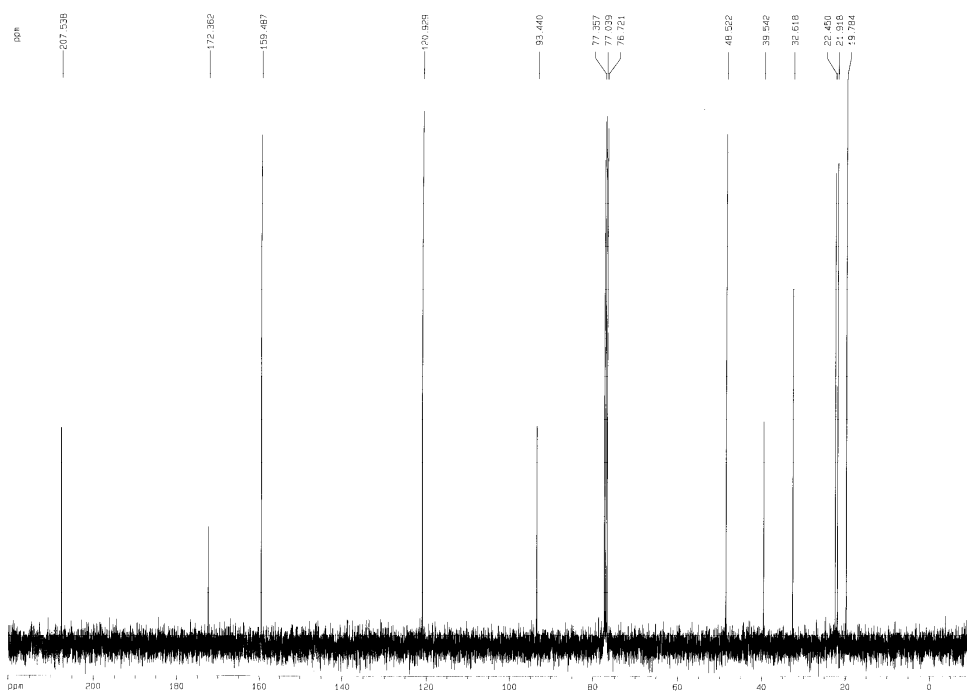
F2 - Acquisition Parameters
Date_ 20070908
Time 0.21
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 8278.146 Hz
FIDRES 0.125314 Hz
AQ 3.598443 sec
RG 40.3
OW 60.400 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MEWIR 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300055 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 21.00 cm
F1P 10.000 ppm
F1 4000.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
H1NRM 0.31250 ppm/cm
HZCM 125.0938 Hz/cm

DPX400 carbon



Current Data Parameters
NAME K05002
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20071212
Time 12.06
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 34
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 3649.1
OW 13.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.09999999 sec
MCREST 0.00000000 sec
MEWIR 0.01500000 sec

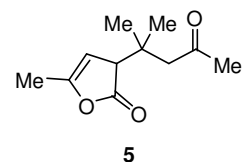
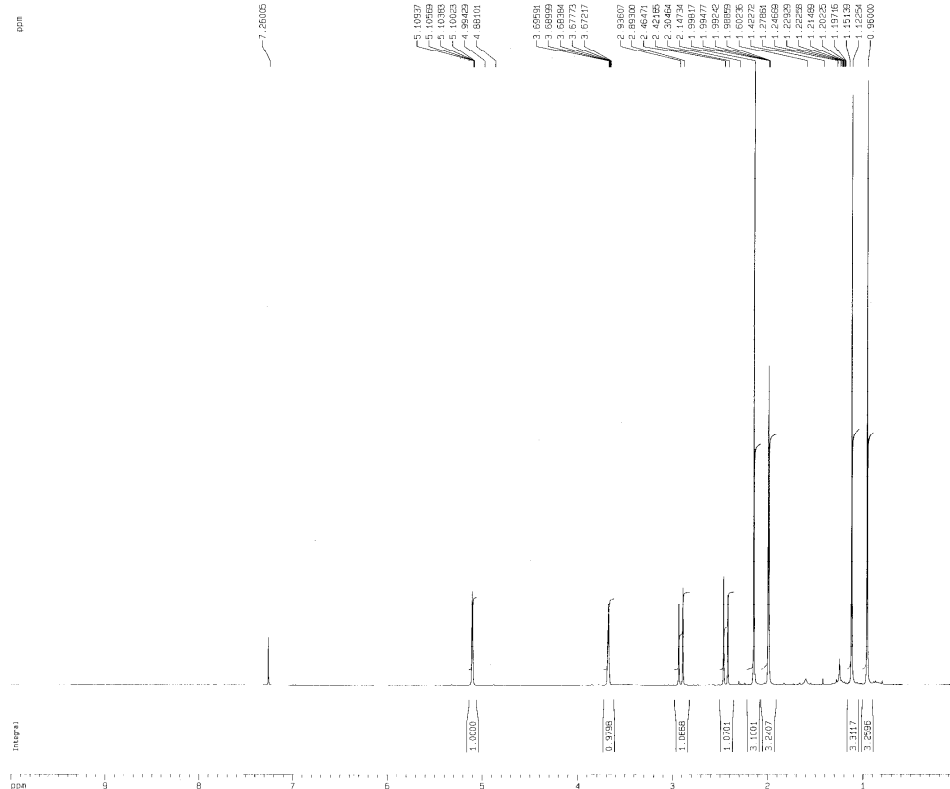
----- CHANNEL f1 -----
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5868886 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0310001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876540 MHz
WDW EN
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 19.00 cm
F1P 220.000 ppm
F1 22129.26 Hz
F2P -10.000 ppm
F2 -1005.86 Hz
H1NRM 7.18750 ppm/cm
HZCM 722.97357 Hz/cm

0.00000000



Current Data Parameters
NAME: AUC00J
EXPNO: 1
PROCNO: 1

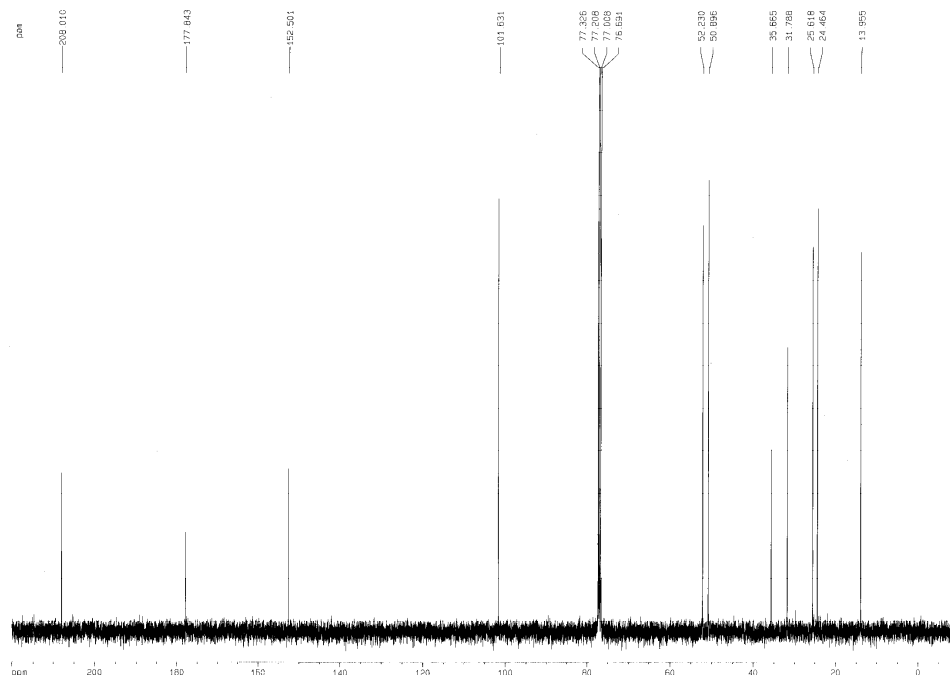
F2 - Acquisition Parameters
Date_: 20071121
Time: 14.18
INSTRUM: spect
PROBHD: 5 mm QNP 1H/13
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 64
DS: 2
SWH: 8278.145 Hz
FIDRES: 0.126314 Hz
AQ: 3.3584243 sec
RG: 143.7
OW: 60.400 usec
DE: 6.00 usec
TE: 300.2 K
D1: 1.00000000 sec
MCREST: 0.00000000 sec
MCWRR: 0.01500000 sec

----- CHANNEL f1 -----
NUC1: 1H
P1: 11.50 usec
PL1: -4.00 dB
SFO1: 400.0324703 MHz

F2 - Processing parameters
SI: 32768
SF: 400.0300517 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00

1D NMR plot parameters
CX: 32.00 cm
CY: 25.00 cm
EXP: 10.000 ppm
F1: 4000.30 Hz
F2: 0.000 ppm
F3: 0.00 Hz
PPMCM: 0.31250 ppm/cm
HZCM: 125.00938 Hz/cm

DPX400 carbon



Current Data Parameters
NAME: AGE003
EXPNO: 2
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20071121
Time: 14.27
INSTRUM: spect
PROBHD: 5 mm QNP 1H/13
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 225
DS: 4
SWH: 26178.010 Hz
FIDRES: 0.398445 Hz
AQ: 1.251075 sec
RG: 15384
OW: 19.100 usec
DE: 6.00 usec
TE: 300.2 K
D1: 2.00000000 sec
D11: 0.03000000 sec
DELTA: 1.88000000 sec
MCREST: 0.00000000 sec
MCWRR: 0.01500000 sec

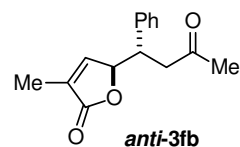
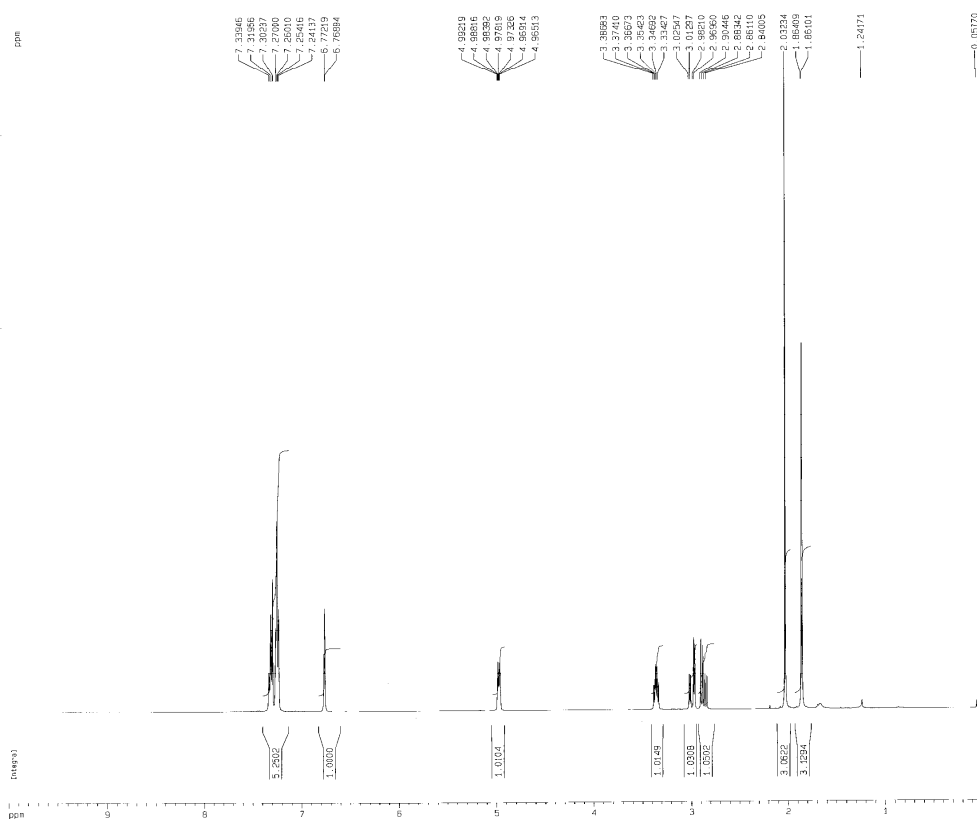
----- CHANNEL f1 -----
NUC1: 13C
P1: 10.00 usec
PL1: -3.50 dB
SFO1: 100.6261800 MHz

----- CHANNEL f2 -----
CPDPRG2: waltz16
NUC2: 1H
PCPD2: 160.00 usec
PL2: -4.00 dB
PL12: 17.00 dB
PL13: 17.00 dB
SFO2: 400.0316001 MHz

F2 - Processing parameters
SI: 32768
SF: 100.6261800 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40

1D NMR plot parameters
CX: 32.00 cm
CY: 15.00 cm
F1: 220.000 ppm
F2: 220.000 Hz
F3: -10.000 ppm
F4: 1000.88 Hz
PPMCM: 7.16150 ppm/cm
HZCM: 722.97537 Hz/cm

DPX400 proton



Current Data Parameters
NAME takashi106 alphaethyl major
F1NAME
PROCNO 1

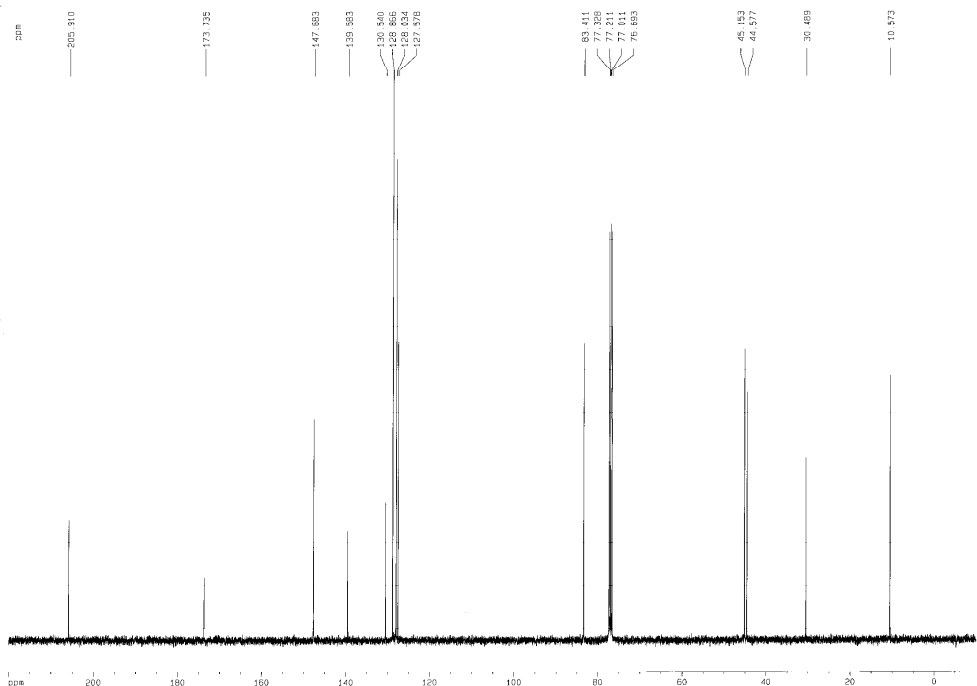
F2 - Acquisition Parameters
Date_ 20091040
Time 15:41
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 8078.146 Hz
FIDRES 0.102314 Hz
AQ 3.9984243 sec
RG 381
DM 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.0000000 sec
d11 0.0000000 sec
MCHEST 0.0150000 sec
HCHW 0.0150000 sec

===== CHANNEL f1 =====
NUC1 13
P1 11.50 usec
PL1 -1.00 dB
SFO1 400.626183 MHz

F2 - Processing parameters
SI 32768
SF 400.130000 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 23.10 cm
FID 16.000 sec
F1 4000.20 Hz
F2 8.000 usec
F3 0.00 Hz
F4 0.00 Hz
F5 0.000000 sec
HCHW 125.00336 Hz/cm

DPX400 carbon



Current Data Parameters
NAME takashi106 alphaethyl major
F1NAME
PROCNO 1

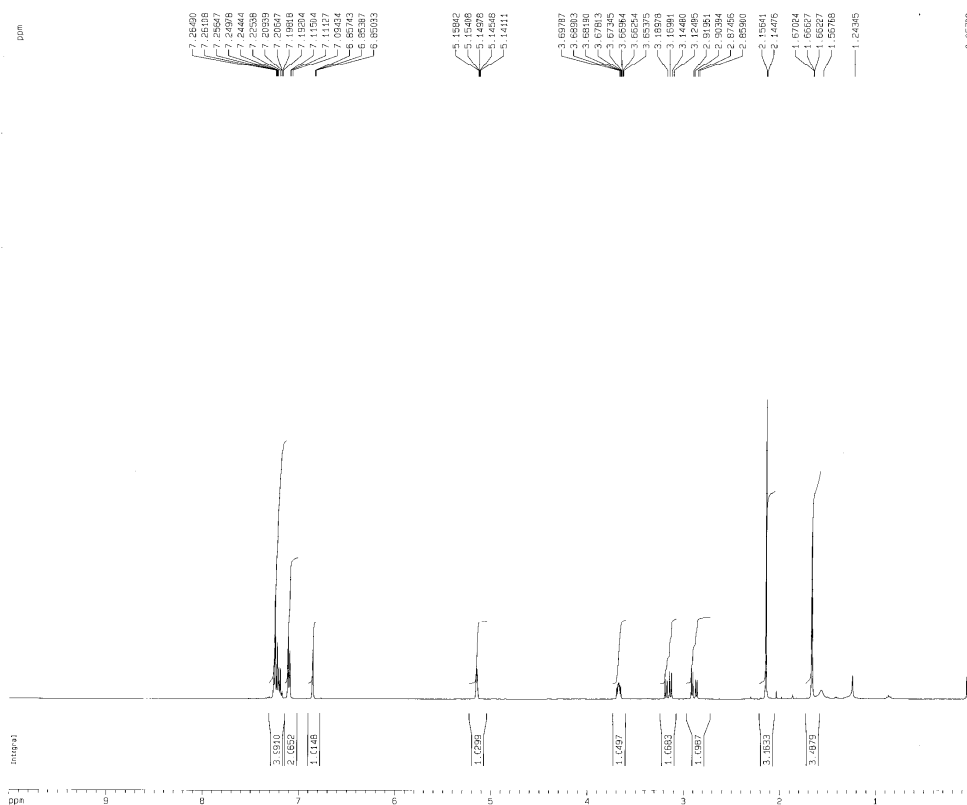
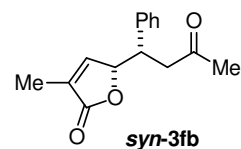
F2 - Acquisition Parameters
Date_ 20091040
Time 20:04
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 384
DS 2
SWH 25176.012 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 409
DM 15.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.0000000 sec
d11 0.0000000 sec
d11.1 0.0000000 sec
MCHEST 0.0150000 sec
HCHW 0.0150000 sec

===== CHANNEL f2 =====
NUC2 13
P2 10.00 usec
PL2 -1.00 dB
SFO2 100.626183 MHz

===== CHANNEL f1 =====
CPDPRG2 waltz16
NUC1 1H
P1 10.00 usec
PL1 0.00 dB
P2 10.00 usec
PL2 0.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0516001 MHz

F2 - Processing parameters
SI 32768
SF 100.626183 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 23.10 cm
FID 16.000 sec
F1 20120.20 Hz
F2 -10.000 usec
F3 1465.00 Hz
F4 7.15750 ppm/cm
HCHW 722.57363 Hz/cm



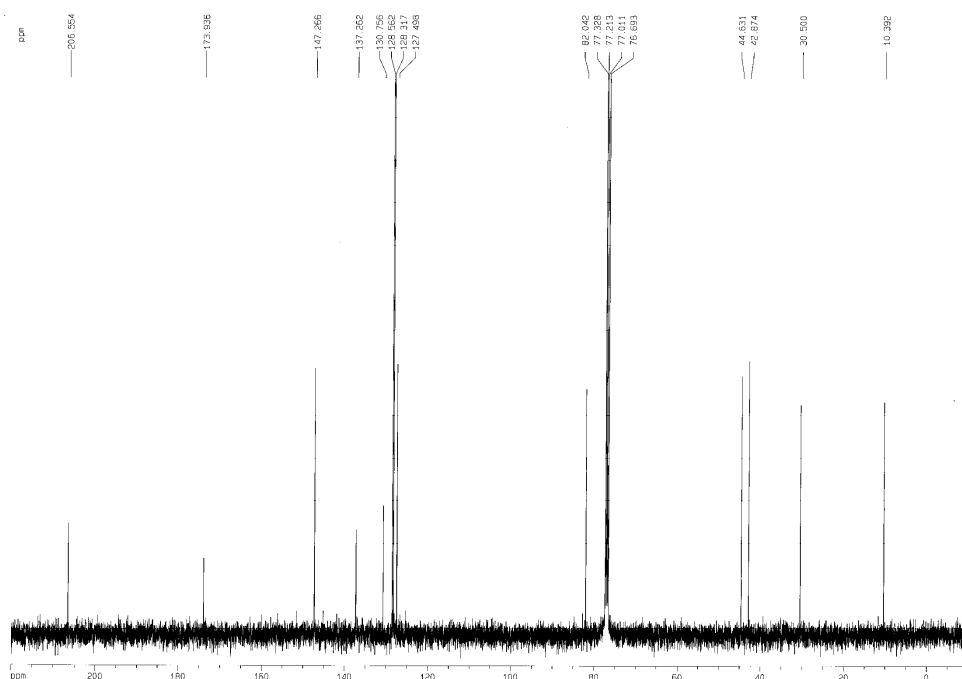
Current Data Parameters
NAME: takashiAM: alphaethyl: minor
COMP: 1
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20091010
Time: 13.57
INSTRUM: spect
PROBHD: 5 mm QNP 1H/13
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 64
DS: 4
SWH: 8278.146 Hz
FIDRES: 0.130314 Hz
AQ: 3.9584243 sec
RG: 327.7
RW: 80.400 kHz
FE: 6.00 usec
TE: 300.2 K
D1: 0.0000000 sec
d11: 0.0000000 sec
MCREST: 0.0150000 sec
MCWID: 0.0150000 sec

----- CHANNEL f1 -----
NUC1: 1H
P1: 11.50 usec
PL1: -4.00 dB
SFO1: 400.0264503 MHz
F2 - Processing parameters
SI: 32768
SF: 400.0264503 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00

1D NMR plot parameters
CX: 35.00 cm
CY: 0.00 cm
F1P: 10.000 ppm
F1: 800.30 Hz
F2P: 0.000 ppm
F2: 0.00 Hz
PUNCH: 3.31250 cm/sec
H1CN: 175.00036 Hz/cm

DPX400 carbon



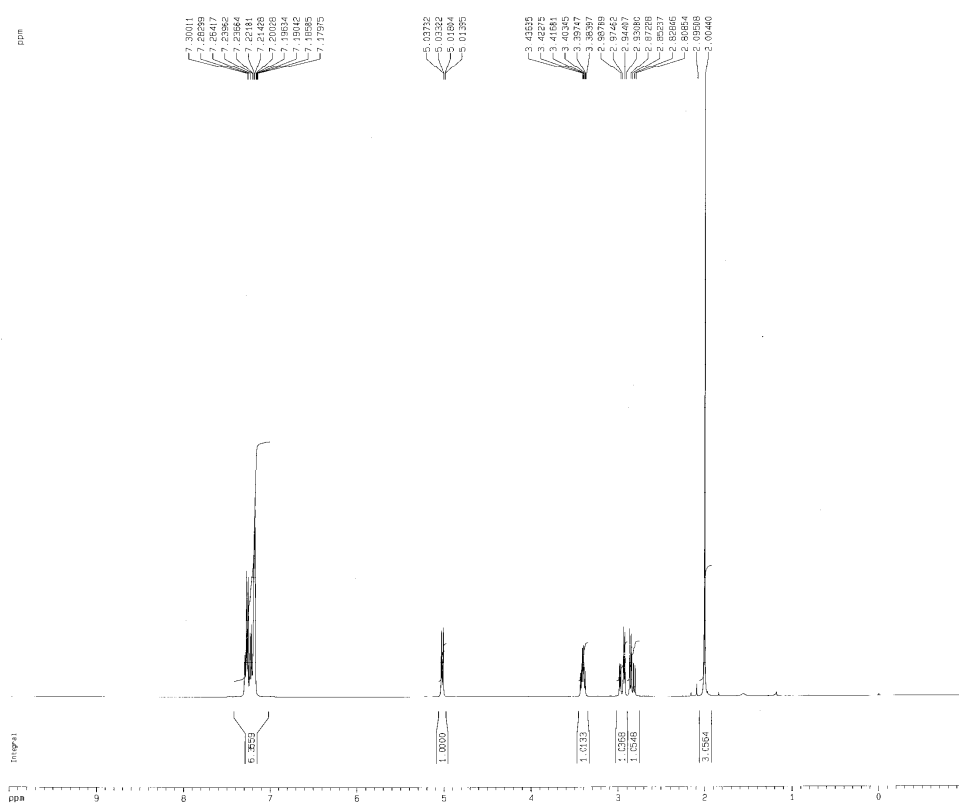
Current Data Parameters
NAME: takashiAM: alphaethyl: minor
COMP: 2
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20091010
Time: 14.55
INSTRUM: spect
PROBHD: 5 mm QNP 1H/13
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 720
DS: 4
SWH: 20160.000 Hz
FIDRES: 0.359440 Hz
AQ: 1.2517875 sec
RG: 519.6
RW: 19.100 kHz
FE: 8.00 usec
TE: 300.2 K
D1: 2.0000000 sec
d11: 0.0000000 sec
MCREST: 0.0000000 sec
MCWID: 0.0150000 sec

----- CHANNEL f1 -----
NUC1: 13C
P1: 10.00 usec
PL1: -3.50 dB
SFO1: 100.6261805 MHz
----- CHANNEL f2 -----
CPDPRG2: waltz16
NUC2: 1H
P2P2P2: 100.00 usec
F2: -4.00 dB
F1F2: 17.00 kHz
F1F2: 17.00 kHz
SFO2: 400.0316501 MHz

F2 - Processing parameters
SI: 32768
SF: 100.6261805 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40

1D NMR plot parameters
CX: 30.00 cm
CY: 10.00 cm
F1P: 220.000 ppm
F1: 223.261 MHz
F2P: -10.000 ppm
F2: -100.626 MHz
PUNCH: 7.18750 cm/sec
H1CN: 722.51257 Hz/cm

CC(=O)C[C@H](Cc1ccccc1)[C@@H]2C=C(Br)C(=O)O2
anti-3fe

```

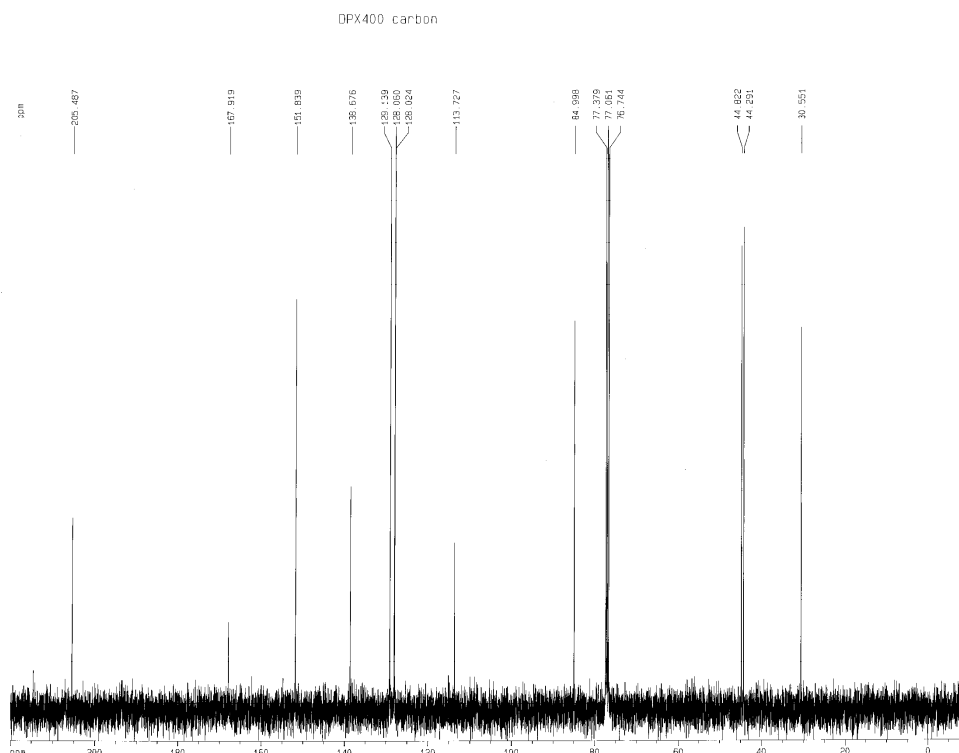
Current Data Parameters
NAME      tosyamide.AM0019
EXPNO     1
PROCNO    1
PROCNAME   1

F2 - Acquisition Parameters
Date_     20090126
Time      14.13
INSTRUM    spect
PROBHD     5 mm QNP 1H/13
PULPROG    zgpg30
TD         65536
SOLVENT    CDCl3
NS          64
DS          2
SWH         8278.146 MHz
FIDRES     0.25314 Hz
AQ          3.9564640 sec
RG          628
INW         50.400 usec
DE          6.000 usec
TE          300.2 K
D1          1.00000000 sec
MCHST1     0.00000000 sec
AQW1       0.01500000 sec

***** CHANNEL f1 *****
NUC1        13
SF          400.146 MHz
WDW          EM
SSB          0
GB           0
PC           1.00

F2 - Processing parameters
Invert plot parameters
CX          32.00 cm
CY          32.00 cm
CZ          10.000 ppm
F1          400.146 MHz
F2          -5000.133 ppm
F3          0.2421699 ppm/MHz
HZ2CM       137.51013 Hz/cm

```



```

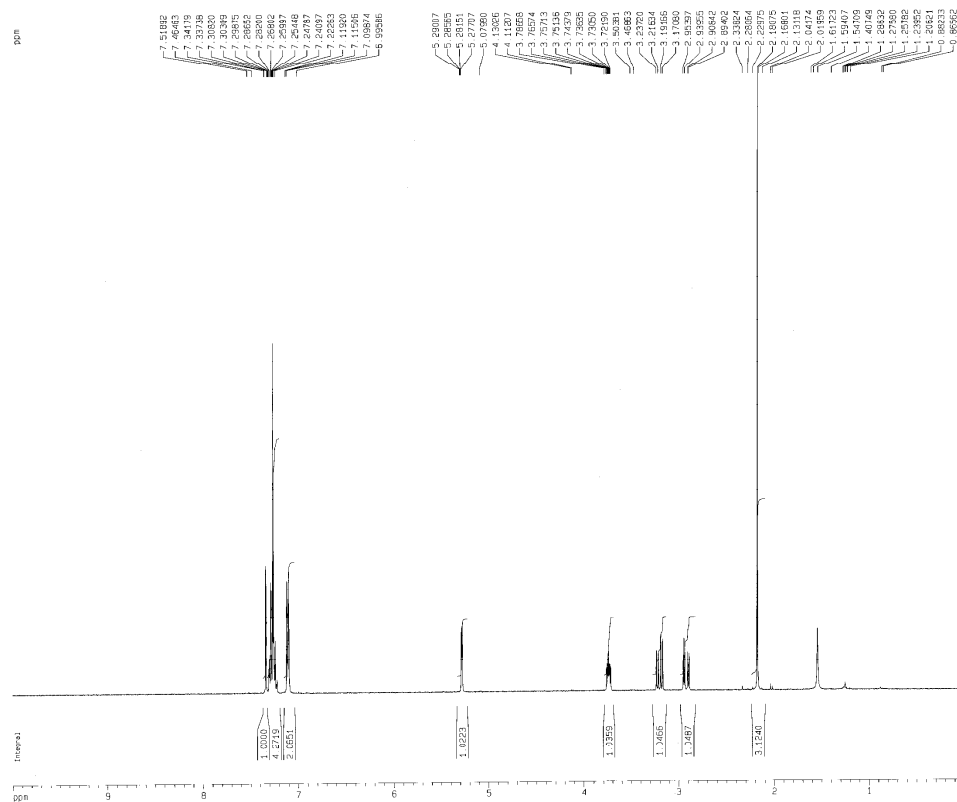
Current Data Parameters
Name      takahashiAK003
EXPNO     4
PROCNO    1

F2 - Acquisition Parameters
Date_      20090916
Time       14:56
INSTRUM    spect
PROBHD     5 mm QNP 1H/1
PULPROG     zgpg30
PCPDPRG2    F2F2B3
SOLVENT     DMSO
NS          63
DS          4
SWH         26178.1014 Hz
AQ          0.399445 sec
FIDRES      1.2571787e-5
RG          6500
AQ          19.100 sec
DQ          6.000 sec
NUC1         1      2.000000000 sec
NUC2         1      0.030000000 sec
TE          1      0.099999998 sec
MAGNET      0.000000000 sec
MPCPRG2     0.015000000 sec
----- CHANNEL f1 -----
NUC1         1H
P1           13C
PL1          -3.50 dB
SFO1         100.5998866 MHz
----- CHANNEL f2 -----
CPDPRG2     waltz16
NUC2         1H
PULPROG     muoco
PCPDPRG2    0.000000000 sec
PL2          -4.00 dB
PL12         17.00 dB
PL13         17.00 dB
SFO2         400.0136101 MHz

F2 - Processing parameters
SI          32768
SF          100.5998871 MHz
WDW         EM
SSB          0
GB          1.00 Hz
PC          1.40

1D NMR plot parameters
CX           32.00 cm
CY           0.00 cm
F1           220.000 ppm
F2           229.199 ppm
F3           120.000 ppm
F4           -100.588 ppm
PCPDPRG2     1.757550 ppm/cm
PCPDPRG2     72.972777 Hz/cm
PCPDPRG2     1.000000 Hz/cm

```



```

Current Data Parameters
NAME      takahashiAMK004
EXPNO     3
PROCNO    1

2D - Acquisition Parameters
Date_     20061113
Time      0.04
INSTRUM    spect
PROBHD     5 mm QNP 1H/13
PULPROG    zgpg30
TD         65536
SOLVENT    CDCl3
AQ         4
DS         2
SM         8278.146 Hz
FIDRES     0.18034 Hz
AQ         3.958460403 sec
RG         817.3
TE         300.2 K
DE         1.00000000 sec
MCHST      0.00000000 sec
HWDW      0.15000000 sec

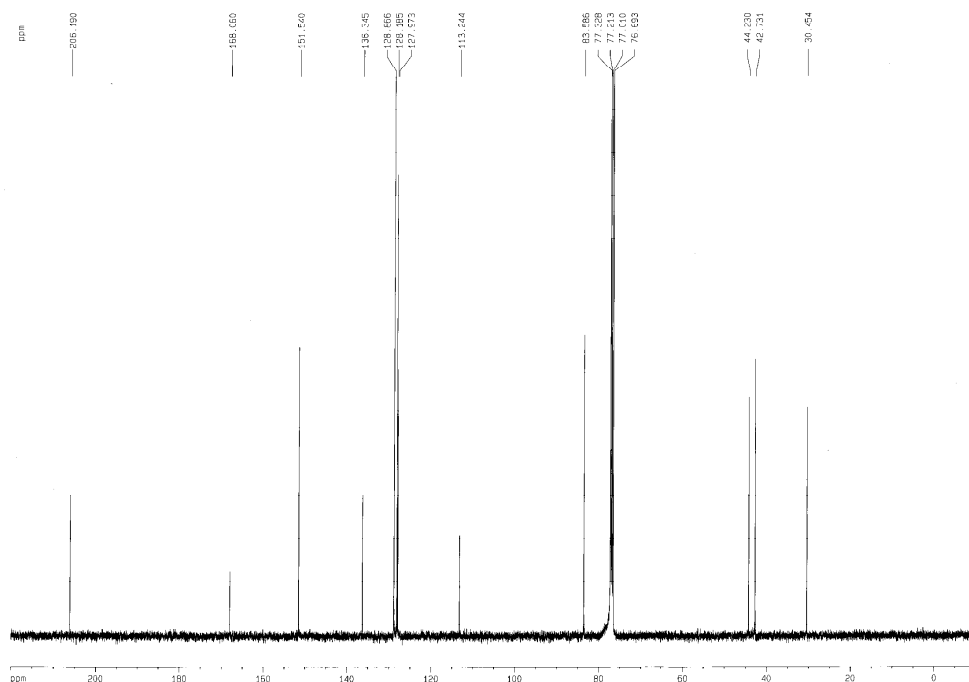
***** CHANNEL f1 *****
NUC1        13
P1          11.50 usec
PL1         -4.00 dB
SF01        400.6384703 MHz

2D - Processing parameters
SI          32768
SF          400.6300000 MHz
WDW         EM
SSB         0
GB          0.30 Hz
PC          1.00

to hsqc print parameters
CX          31.00 cm
CY          20.00 cm
F1          10.000 ppm
F2          400.000 MHz
P2          0.000 ppm
PC          1.00
NUC2        13
H2ZM       0.31800000 g/g
H2ZM2      125.00938 Hz/cm

```

DPX400 carbon



```
Current User Parameters
NAME      takahashiAKM004
EXPNO     1
PROCNO    1

Data - Acquisition Parameters
Date_     20090514
Time      0.00
INSTRUM   spect
PROBHD    5 mm QNP 1H/13
PULPROG   zgpg30
TD         65536
AQ         0.0030
SOLVENT   DMSO
NS         10143
DS         4
SWH        25178.10 Hz
FIDRES     0.399445 Hz
AQRES      1.2517578 sec
RG          320
RG2         19.180 sec
DE          0.65 sec
TE         300.2 K
DW          2.0000000 sec
d11         1.0000000 sec
DELTA      1.0000000 sec
DECORST    0.0000000 sec
NUC1PROG   0.0100000 sec

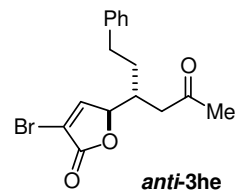
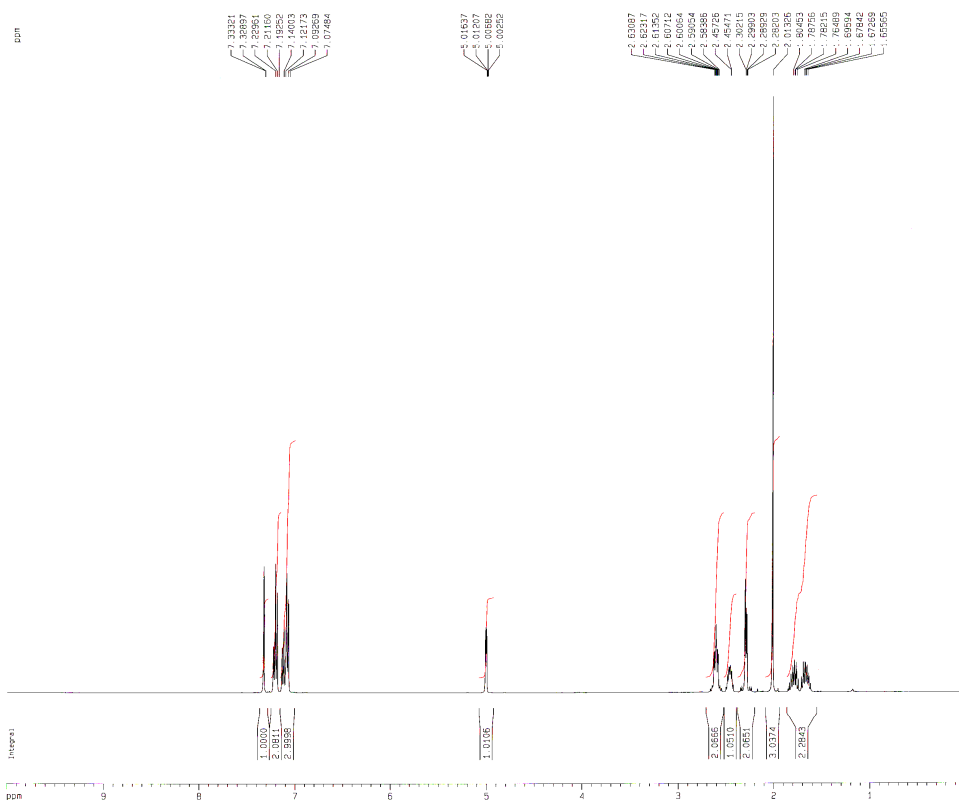
***** CHANNEL f1 *****
NUC1       13C
P1          13C
SF01       100.50688 MHz

***** CHANNEL f2 *****
CFREQPRG   waltz16
NUC2        H1
PCPD2       4.000000 sec
PL1         -4.00 dB
PL2         17.00 dB
PL3         17.00 dB
SF02       400.0150001 MHz

Data - Processing parameters
SI          32768
SF          100.50785230 MHz
WDW         EM
SSB         0
GB          1.00 Hz
PC          1.40

10 peak dipole parameters
CX          32.00 cm
CY          19.00 cm
FZ          220.00 cm
F1          220.00 Hz
F2          -10.000 ppm
F3          -1000.00 Hz
H1ZON      7.16150000 ppm/cm
H2ZON      792.97507 Hz/cm
```


CPYX000 PL 0001



Current Data Parameters
NAME takahashiAK001
EXPNO 1
PROCNO 1

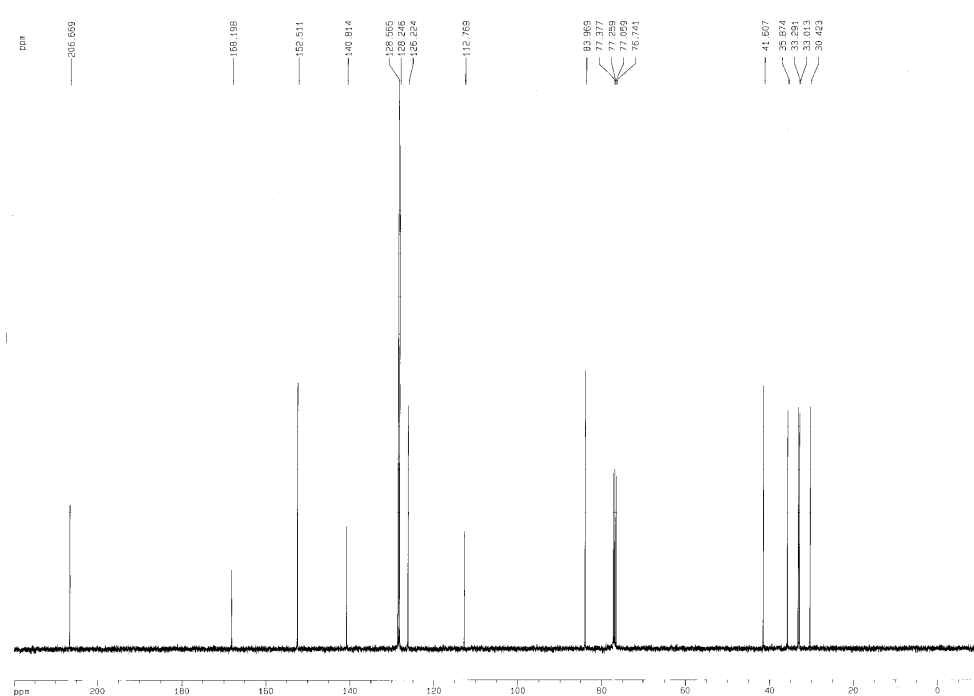
F2 - Acquisition Parameters
Date_ 20090508
Time 20.00
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
VS 2
DS 2
SWH 8278.146 Hz
FIDRES 0.136314 Hz
AQ 3.9584243 sec
RG 101.6
DW 50.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCHST 0.00000000 sec
MCWK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300348 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 0.00 cm
FJP 10.000 ppm
F1 4000.30 Hz
F2 0.000 ppm
F3 0.00 Hz
PRMCM 0.31250 ppm/cm
HZCM 125.00938 Hz/cm

DPX400 carbon



Current Data Parameters
NAME takahashiAK001
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090508
Time 20.00
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
VS 154
DS 4
SWH 26170.910 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 6502
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
DELTA 1.89999998 sec
MCHST 0.00000000 sec
MCWK 0.01500000 sec

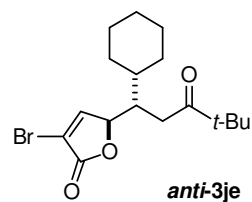
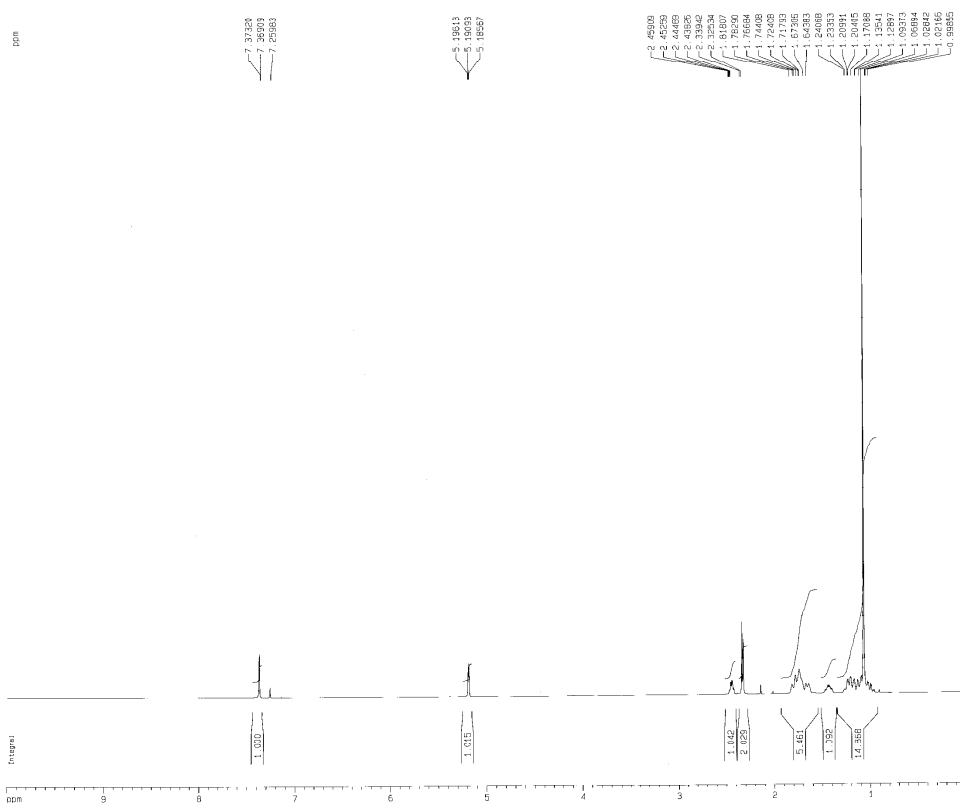
===== CHANNEL f1 =====
NUC1 13C
P1 19.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

===== CHANNEL f2 =====
CPROG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5978284 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 19.00 cm
FJP 220.000 ppm
F1 4000.30 Hz
F2 -10.000 ppm
F3 -1005.88 Hz
PRMCM 7.018750 ppm/cm
HZCM 722.97363 Hz/cm

DPX400 1H 13C



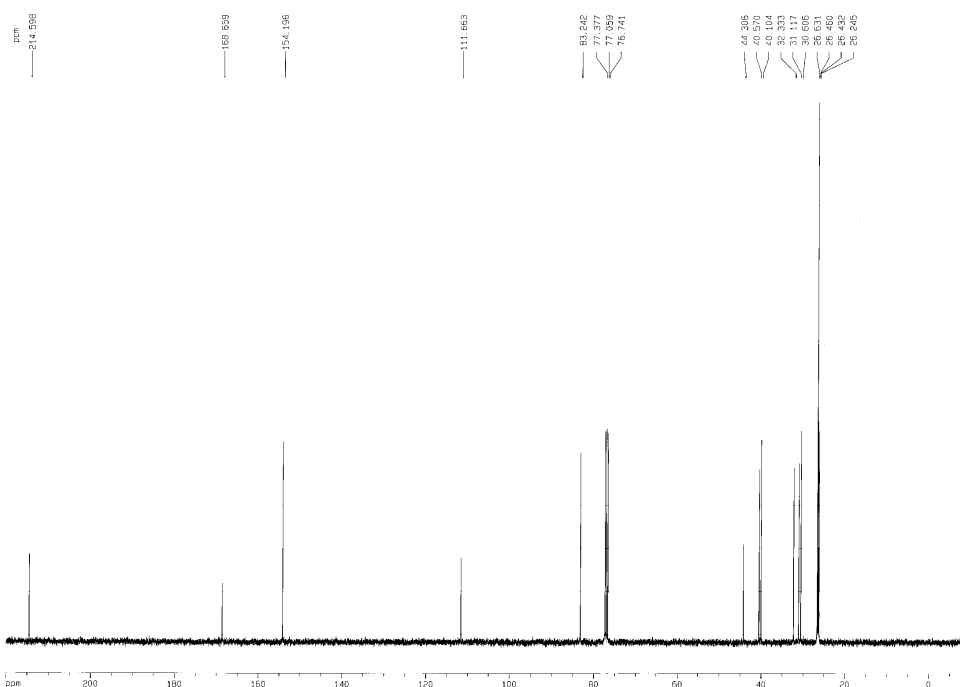
Current Data Parameters
NAME Lokahashi AM009
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090130
Time 14.41
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 101.6
DA 60.400 usec
DE 5.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRR 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0364793 MHz
F2 - Processing parameters
SI 32768
SF 400.030065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 21.00 cm
F1P 10.000 ppm
F1 4000.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPHMC 0.31260 ppm/cm
HZCM 125.00938 Hz/cm

DPX400 carbon



Current Data Parameters
NAME Lokahashi AM009
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090130
Time 14.45
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 156
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 5168.6
DA 19.100 usec
DE 5.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRR 0.01500000 sec

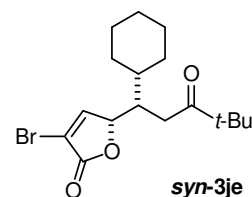
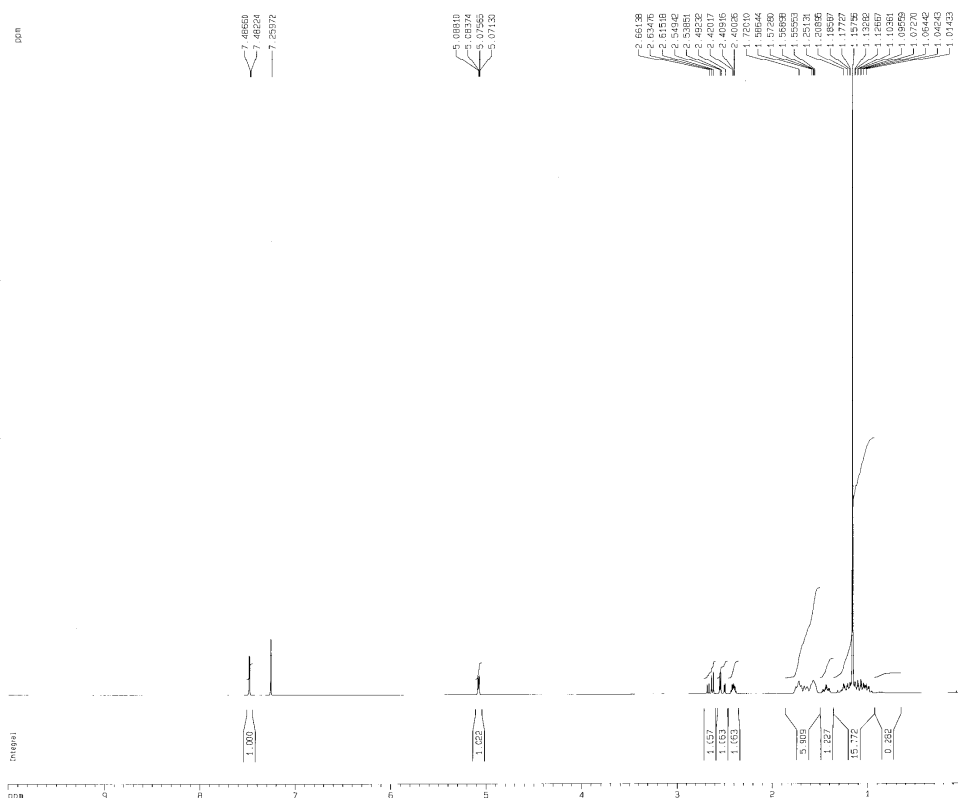
===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986866 MHz

===== CHANNEL f2 =====
CPROG2 waltz16
NUC2 1H
P2P2P 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0310001 MHz

F2 - Processing parameters
SI 32768
SF 100.587628 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 19.00 cm
F1P 220.000 ppm
F1 22129.28 Hz
F2P -10.000 ppm
F2 -1005.88 Hz
PPHMC 7.18750 ppm/cm
HZCM 722.97357 Hz/cm

DPX400 proton



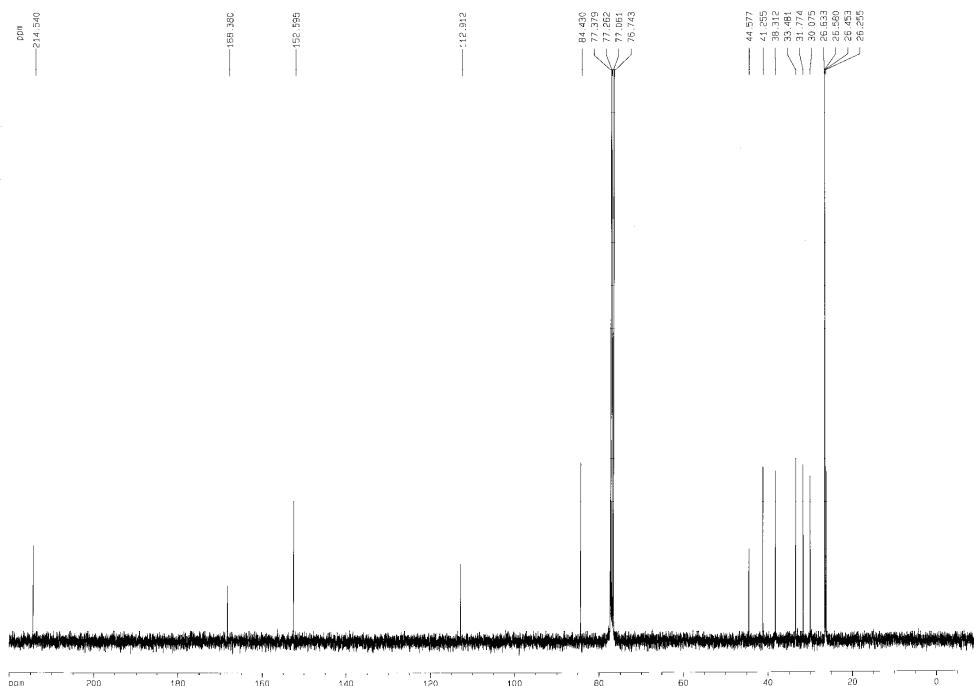
Current Data Parameters
NAME takahashiAK010
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090212
Time 20.19
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 54
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.5904243 sec
RG 287.4
OW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

----- CHANNEL F1 -----
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300067 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 21.00 cm
F1P 10.000 ppm
F1 4000.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PUMCM 0.31250 ppm/cm
HZCM 125.00938 Hz/cm

DPX400 carbon



Current Data Parameters
NAME takahashiAK010
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090212
Time 20.43
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 716
DS 4
SWH 8278.146 Hz
FIDRES 0.299445 Hz
AQ 1.2517875 sec
RG 287.4
OW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.88999998 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

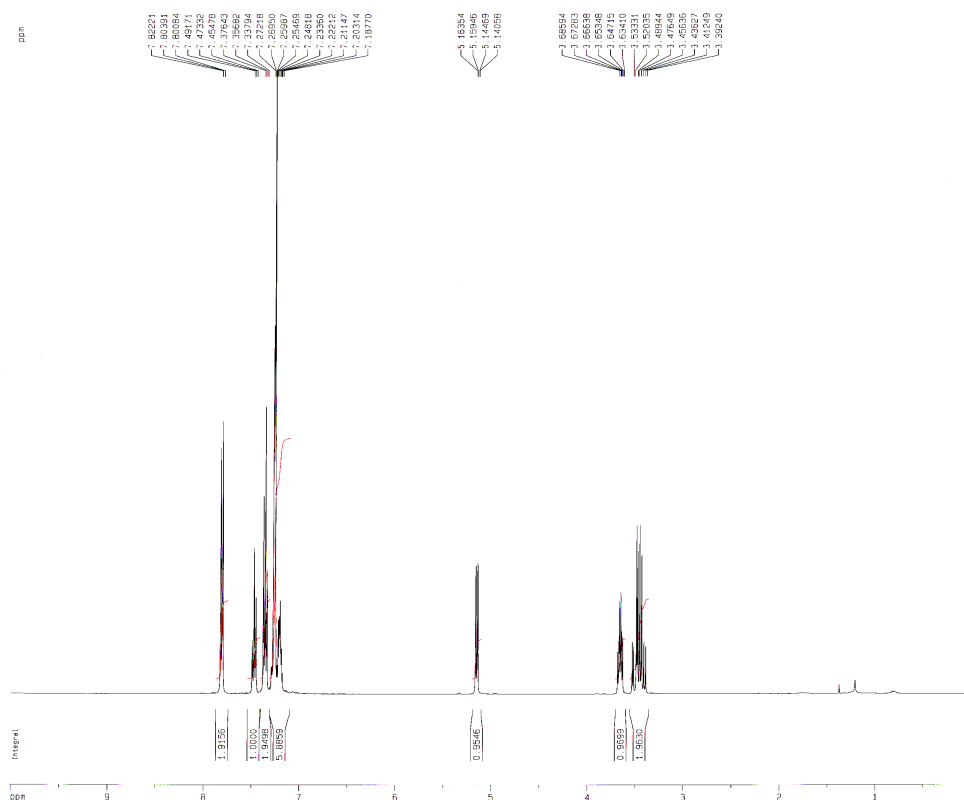
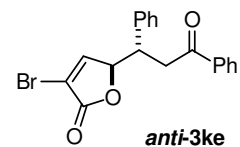
----- CHANNEL F1 -----
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

----- CHANNEL F2 -----
CPDPRG2 waltz16
NUC2 1H
PROG2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0346001 MHz

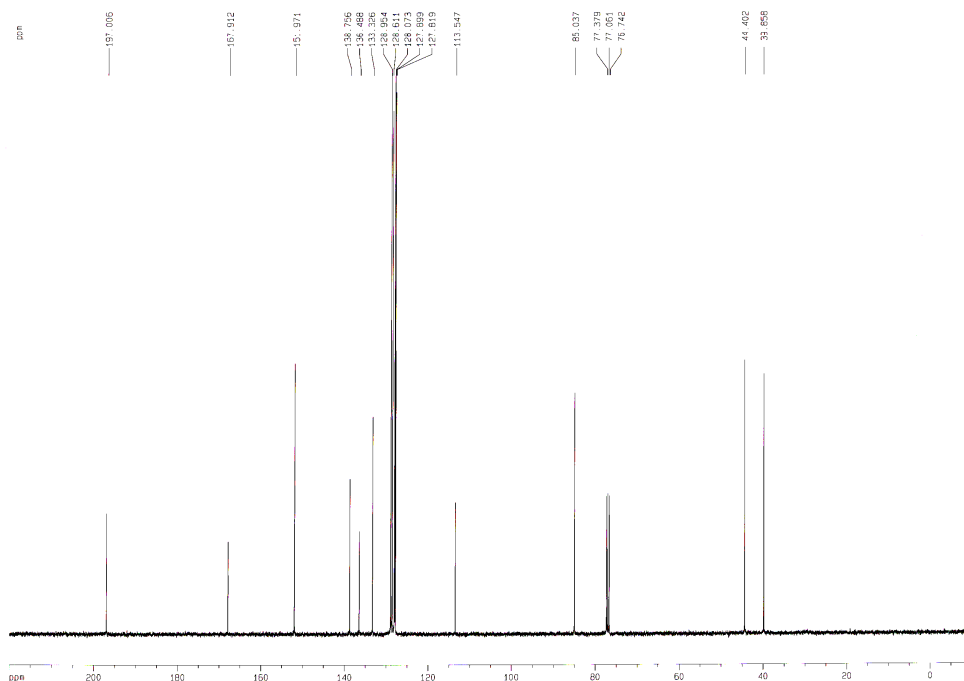
F2 - Processing parameters
SI 32768
SF 100.5876180 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 19.00 cm
F1P 220.000 ppm
F1 22129.26 Hz
F2P -10.000 ppm
F2 -1005.88 Hz
PUMCM 7.18750 ppm/cm
HZCM 722.97351 Hz/cm

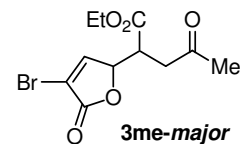
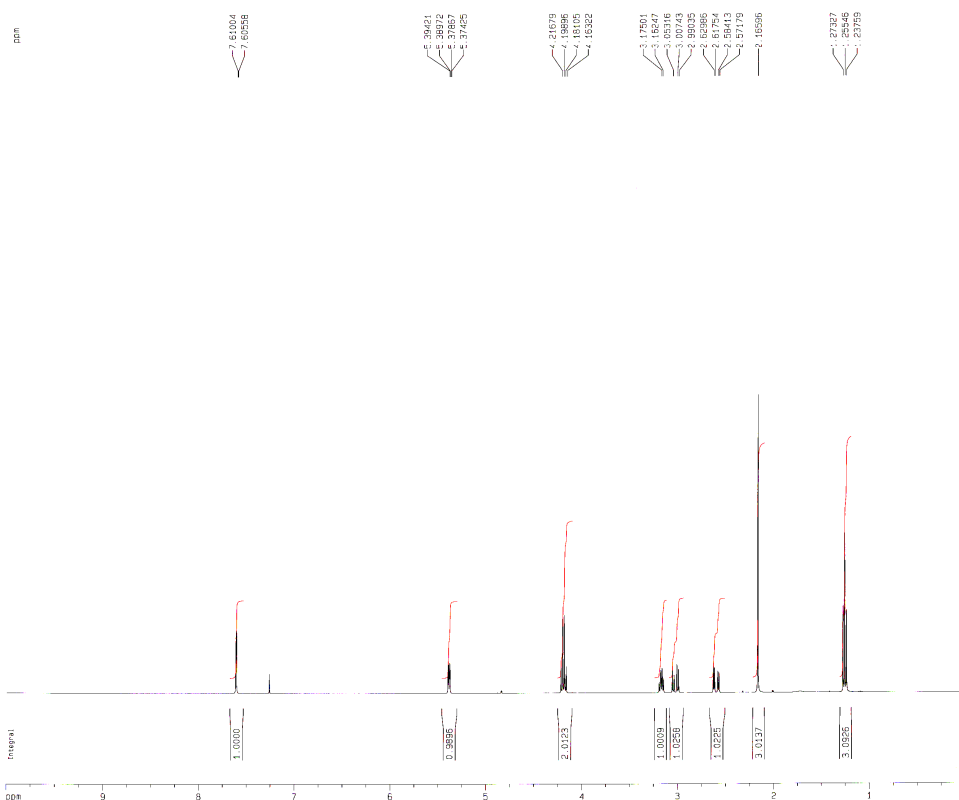
JYX400 1H/13C



DPX400 carbon



DPX400 proton



Current Data Parameters
NAME takahashiANK023
EXPNO 1
PROCNO 1

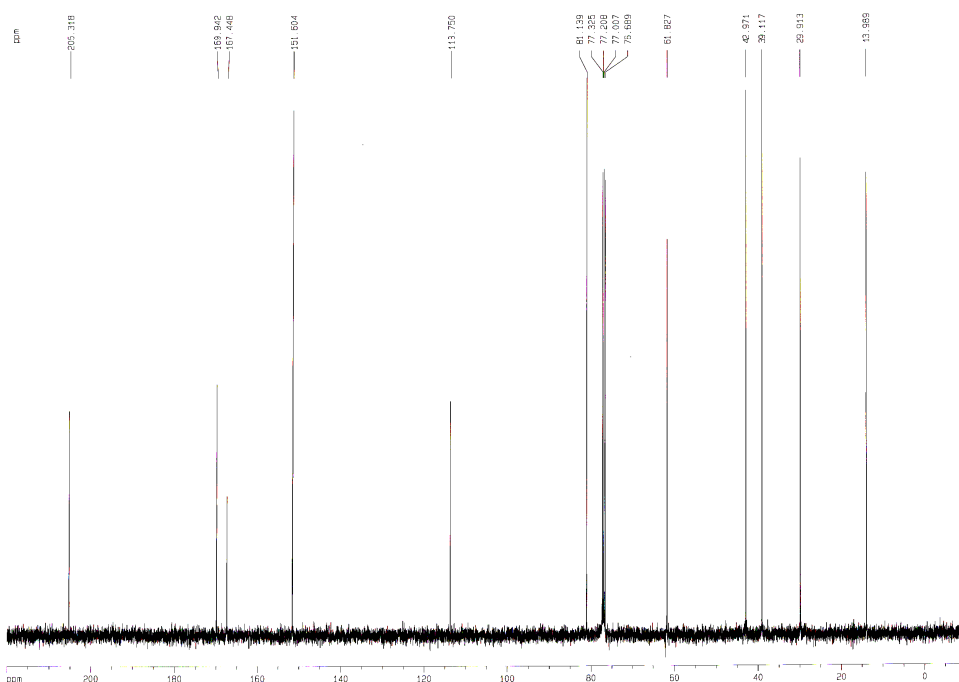
F2 - Acquisition Parameters
Date_ 20090503
Time 13.34
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 128
DM 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCHST 0.00000000 sec
MCHRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300052 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 0.00 cm
F1P 10.000 ppm
F1 4000.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.31250 ppm/cm
HZCM 125.00938 Hz/cm

DPX400 carbon



Current Data Parameters
NAME takahashiANK023
EXPNO 2
PROCNO 1

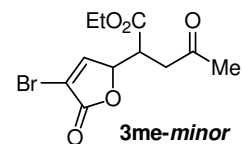
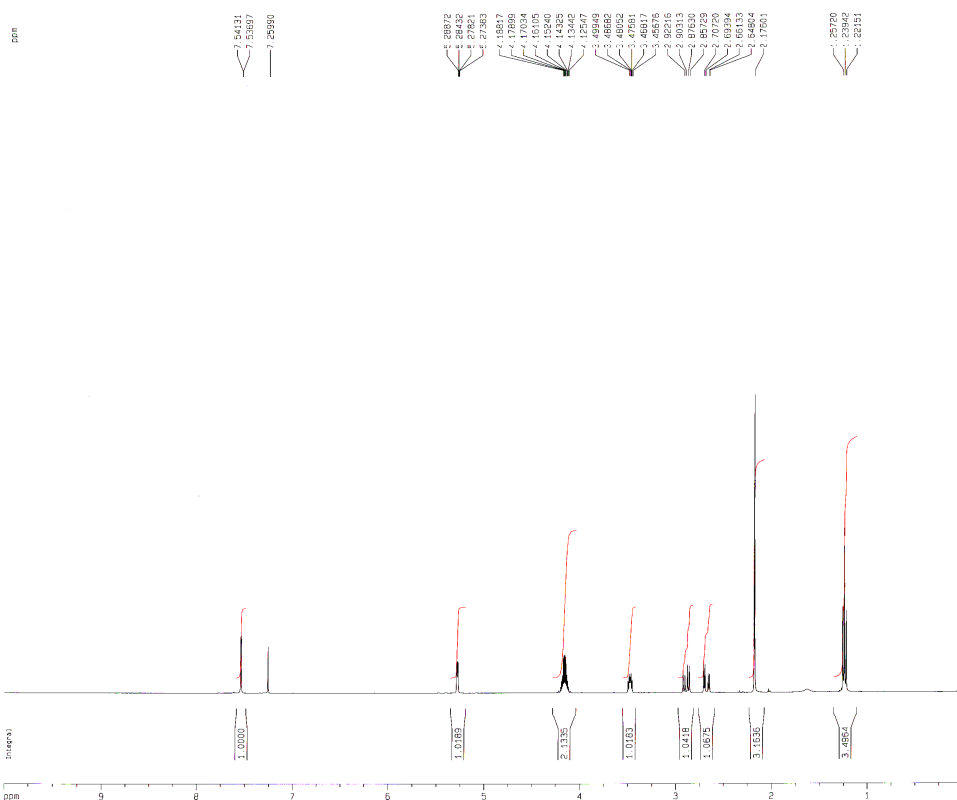
F2 - Acquisition Parameters
Date_ 20090503
Time 13.43
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 135
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2518775 sec
RG 1824.5
DM 19.100 usec
DE 5.00 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.55999998 sec
MCHST 0.00000000 sec
MCHRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 -13.50 dB
SFO1 100.5986860 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876286 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 19.00 cm
F1P 220.000 ppm
F1 221.2323 Hz
F2P -10.000 ppm
F2 -1005.88 Hz
PPMCM 7.18750 ppm/cm
HZCM 722.97363 Hz/cm



```

Current Data Parameters
NAME      takahashiAMQ24
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20090603
Time      13:57
INSTRUM    spect
PROBHD     5 mm QNP 1H/13
PULPROG    zgpg30
TD          65536
SOLVENT    DMSO
NS          16
DS          2
SWH         8278.146 Hz
FIDRES     0.12614 Hz
AQ          3.9564341 sec
RG           4
DE          60.400 usec
TE          6.00 usec
AQE         300.00 %
RG1         1.00000000 sec
RG2         0.00000000 sec
RG3         0.01500000 sec

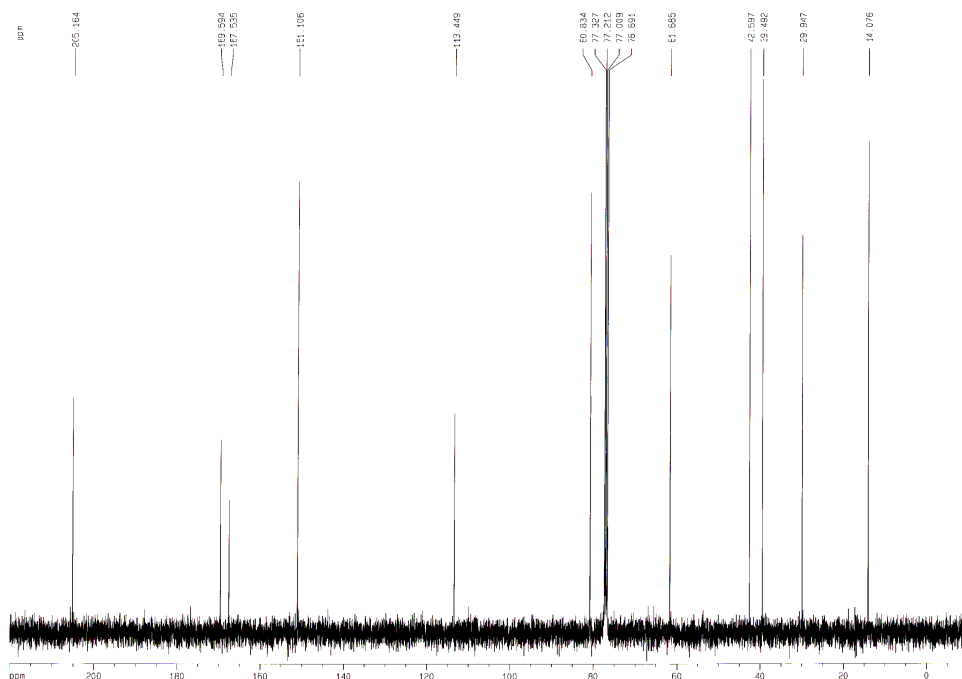
***** CHANNEL f1 *****
NUC1        13
P1           1.50 usec
PL1          -4.00 dB
SFO1        400.1324703 MHz

F2 - Processing parameters
SI          32768
SF          400.1300000 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00

1D NMR plot parameters
CX           30.00 cm
CY           10.000 ppm
F2           4000.000 Hz
F3           60000.000 Hz
FZ           0.000 Hz
PCENH        0.3150 ppm
H2Z          125.00998 Hz/c

```

DPX400 carbon



```

Current Data Parameters
NAME      lakshaniAMK024
EXPNO     2
PROCNO    1

=====
F2 - Acquisition Parameters
DATE_     20090603
TIME      14:20
INSTRUM    spect
PROBHD     5 mm QNP 1H/13
PULPROG    zgpg30
RG          2.000000
AQ         0.0536
SOLVENT    DMSO
NS          400
DS          4
SWH         26178.70 Hz
FIDRES     0.39940 Hz
AQRES      1.250000 sec
RG          4.0
DE          19.000 usec
TE          6.00 usec
TE2        300.2 K
NUC1        1
NUC2        13
P1          2.0000000 sec
d11         0.0300000 sec
d12         1.0000000 sec
MKREST     0.0500000 sec
MKFMC      0.0150000 sec

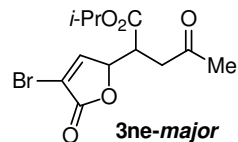
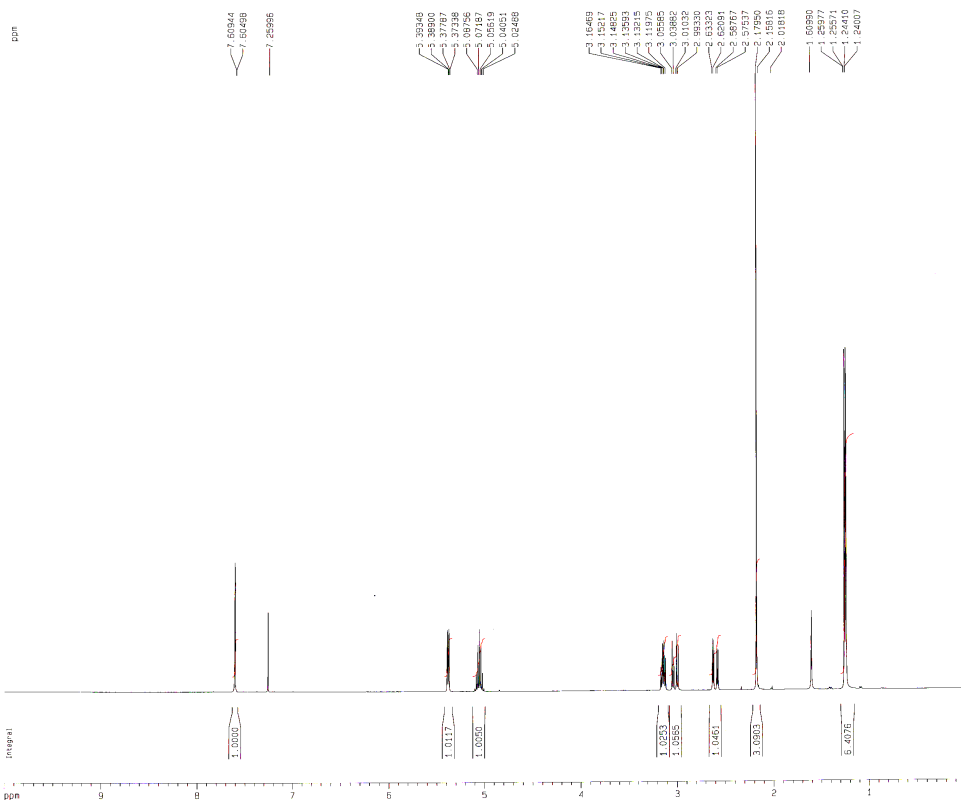
=====
===== CHANNEL 1 =====
NUC1        1H
P1          13C
SFO1        100.508868 MHz

=====
===== CHANNEL 2 =====
CPDPRG2     waltz16
NUC2        1H
P2          1H
PR2         4.0000 usec
SFO2        400.136061 MHz
PL12        17.00 dB
PL13        17.00 dB
SFO2        400.136061 MHz
F2P2        -50.000000 MHz
NDW         10.000
SSB         0.00 Hz
LB          3.00 Hz
PC          1.40

=====
F2 - Processing Parameters
CY          32.00 cm
CX          32.00 cm
CY          220.000 cm
F2P1        220.220 MHz
F2P2        -10.000 ppm
F2P3        -10.000 ppm
PCMDW       700.97555 MHz
H2O2        722.97557 MHz

```

DPX400 1H/13C



Current Data Parameters
NAME takahashi/AM021 019
EXPNO 4
PROCNO 1

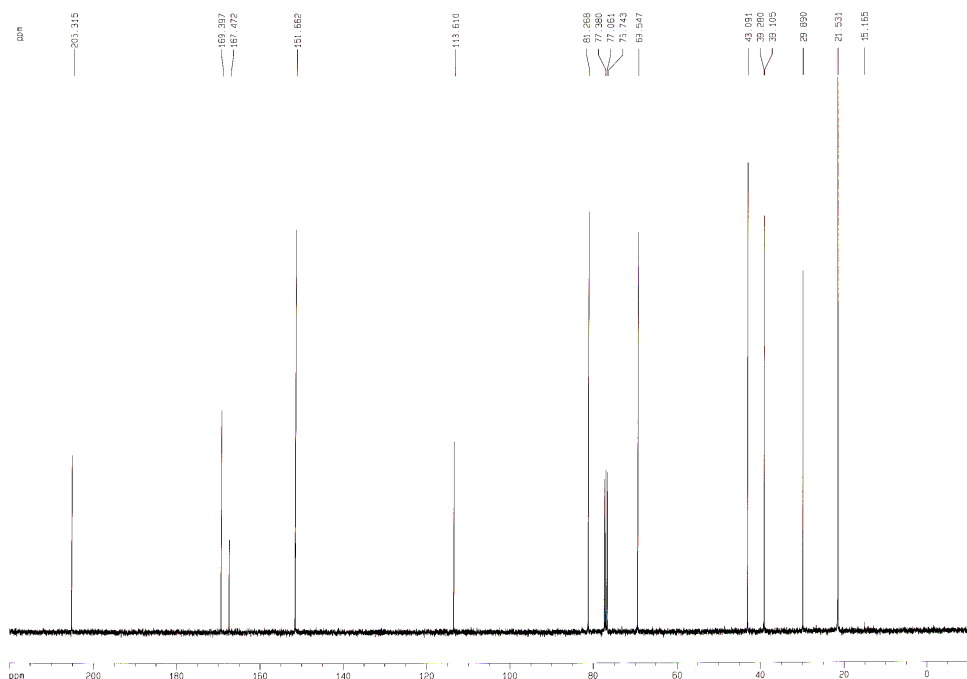
F2 - Acquisition Parameters
Date_ 20090507
Time 17.38
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCHST 0.00000000 sec
MCHRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300005 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 21.00 cm
F1P 10.000 ppm
F1 4000.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPHMC 0.31250 ppm/cm
HZCM 125.00938 Hz/cm

DPX400 carbon



Current Data Parameters
NAME takahashi/AM021
EXPNO 5
PROCNO 1

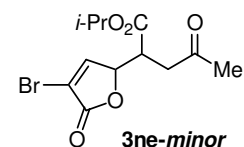
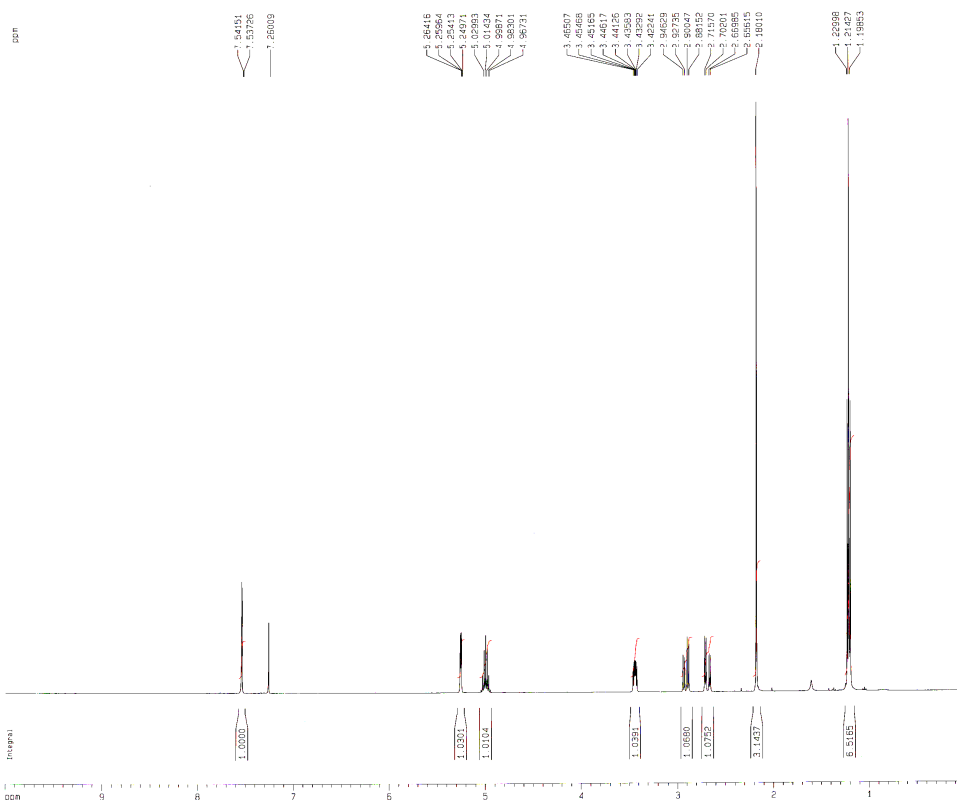
F2 - Acquisition Parameters
Date_ 20090507
Time 18.55
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4
DS 1
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2518785 sec
RG 6192
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
D114 1.00000000 sec
MCHST 0.00000000 sec
MCHRK 0.01500000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5966085 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5970292 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 19.00 cm
F1P 220.000 ppm
F1 22129.28 Hz
F2P -10.000 ppm
F2 -1005.88 Hz
PPHMC 71.15780 ppm/cm
HZCM 722.97363 Hz/cm



```

Current Data Parameters
NAME      takahashiAM020
EXPNO     1
PROCNO    1

2 - Acquisition Parameters
Date_     20090908
Time      13:54
INSTRUM    spect
PROBHD     5 mm QNP 1H/13
PULPROG    zg30
SOLVENT    DMS
SOLVENT2   CDCl3
NS          8
DS          2
SWH         8279.146 MHz
FIDRES      0.126314 Hz
AQ          3.9584640 sec
RG          64
DE          50.400 usec
TE          6.00 usec
TE2         300.2 K
T1          0.0000000 sec
T1RHO       0.0000000 sec
T2          0.0500000 sec
T2RHO       0.0500000 sec

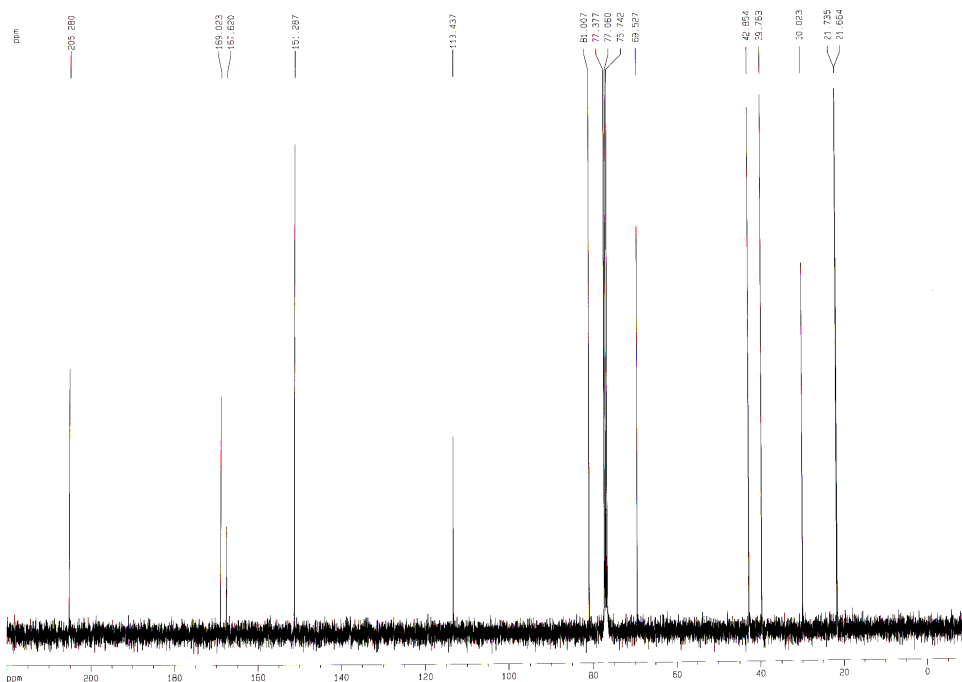
----- CHANNEL f1 -----
NUC1        13
P1          11.50 usec
PL1         -4.00 dB
SFO1        400.0234703 MHz

2 - Processing parameters
SI          32768
SF          400.0300000 MHz
WDW          EM
SSB          0
GB          0.35 Hz
PC          1.00

10 MHz parameters
CX          30.00 cm
CY          0.00 cm
F1P         10.000 ppm
F2P         400.011 MHz
F3P         0.000 MHz
Q           0.000 ppm
R           0.000 Hz
H1ZM        125.00938 Hz/cm

```

DPX400 carbon



```

Current Data Parameters
NAME          ta-aquaria04m20
EXPNO         1
PROCNO        1

F2 - Acquisition Parameters
Date_         20090908
Time          14 23
INSTRUM       spect
PROBHD        5 mm QNP 1H/1
PULPROG       zgpg30
RG            655.56
SOLVENT       DMSO
LQ            CCCL3
NS            491
DS            2
SWH            25170.181 MHz
FIDRES        0.399445 Hz
AQ            1.25170181 sec
RG            655.56
DE            19.100 usec
DM            0.00 usec
TE            300.2 K
DW            2.00000000 sec
D1            0.1 sec
D11           0.189999999 sec
DELTA         0.00000000 sec
WEIGHT        1
WORKR         0.01500000 sec

***** CHANNEL f1 *****
NUC1           13C
P1             100
SFO1          100.599898 MHz

***** CHANNEL f2 *****
NUC2           1H
PCPD0         14.00 usec
P1            -4.00 usec
P2            17.00 dB
PL12          17.00 dB
PL13          17.00 dB
SFO2          400.0136101 MHz

F2 - Processing parameters
PC            1
SI            32768
AQ            100.5978700 MHz
HDW           0.0
SSB           0
GB            0.1 Hz
PC            1.40

ID NMR parameters
CX            32.00 cm
CY            19.00 cm
F1           220.255 MHz
F2           221.255 MHz
F2P           -10.000 MHz
F2P2          720.995 MHz/cm
F2P3          7.1857500 MHz/cm
HDOM          1.00

```

100



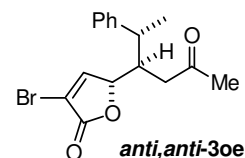
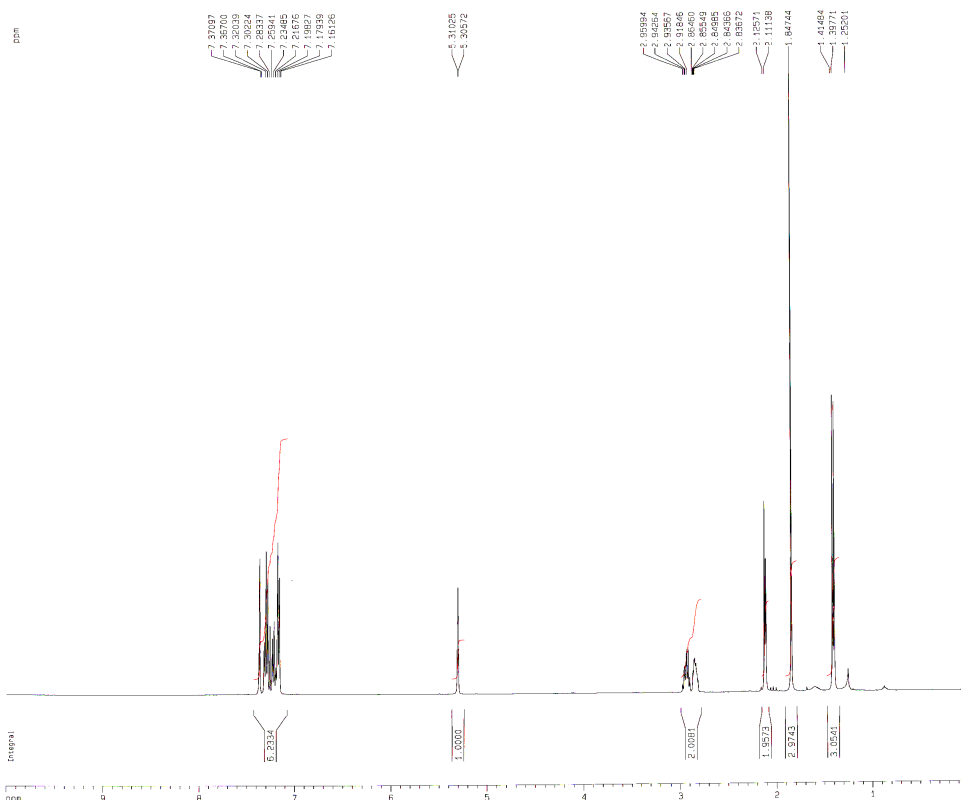
anti,syn-3oe

```

1D NMR plot parameters
CX              32.00 cm
CY              19.00 cm
F1P             220.000 ppm
F1              22129.26 Hz
F2P             -10.000 ppm
F2              -1005.88 Hz
PPMCM           7.1875 ppm/cm
HZCM            72.97357 Hz/cm

```


0207



```

Current Data Parameters
NAME takahashiA00104
EXNO 1
PROBNO 1

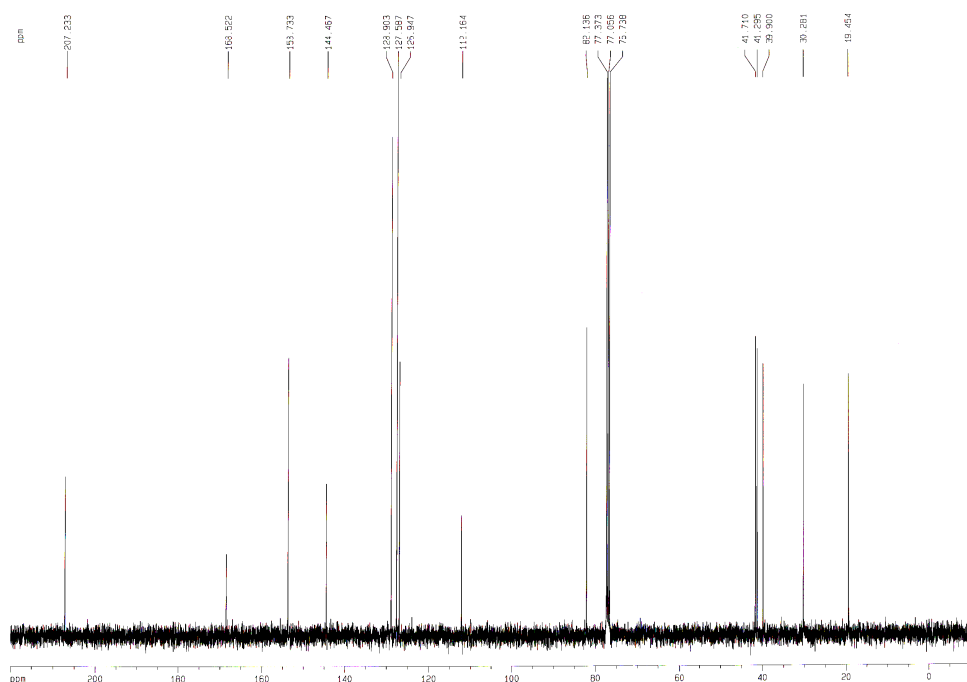
Date - Acquisition Parameters
F2 - 20090129
TIME 12.00
INSTRUM spect
PROBNO 5 mm QNP 1H/13
13CPRG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
AQ 8278.146 sec
FIDRES 0.126314
F2 3.9564440 MHz
DE 160.400 usec
UN 5.00 usec
TE 300.2 K
D1 1.00000000 sec
MCRST 0.00000000 sec
MCRPR 0.01500000 sec

***** CHANNEL 1 *****
MUC1
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.324703 MHz

F2 - Processing parameters
SI 32768
SF 400.3000000 MHz
WDW EM
SSB 0
GB 0
PC 1.00
H1 NMR data parameters
CX 31.00 cm
CY 31.00 cm
F1P 10.000 ppm
F1 400.141 MHz
F2 100.620 ppm
F2 0.00 MHz
F3 0.141 MHz
H1N1 125.000000 MHz

```

DPX400 carbon



```

Current Data Parameters
Name takahashiAM014
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090929
Time 17:07
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
SOLVENT DMSO
NS 131
DS 4
SWH 26176.010 MHz
FIDRES 0.395445 Hz
AQ 1.2517370 sec
RG 8192
DE 19.000 usec
O1 300.2 K
O2 1.0000000 sec
O3 0.0300000 sec
P1 1.8000000 sec
P1A 1.8000000 sec
PC 1.0000000 sec
MCWPRG 0.0150000 sec

----- CHANNEL f1 -----
NUC1 13C
P2 13C
PC2 100.608886 MHz
SFO2 400.0136001 MHz

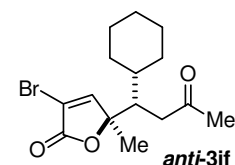
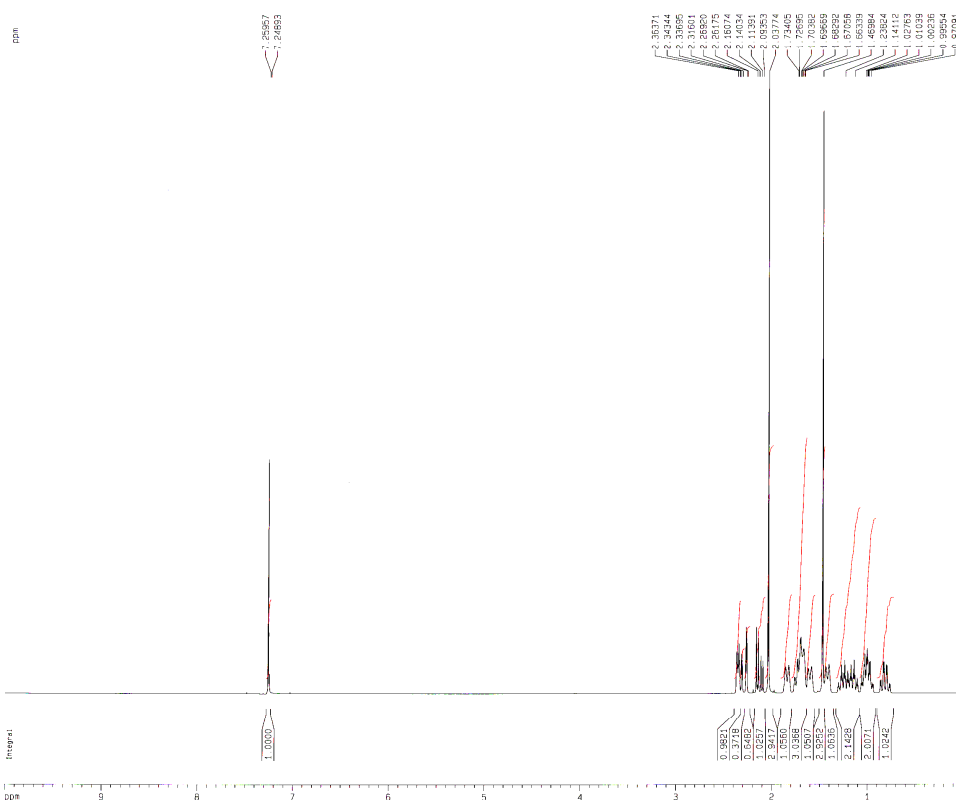
----- CHANNEL f2 -----
NUC2 1H
PC2 1H
PC2P2 100.000 usec
P1 4.0000000 sec
P2 17.000 db
PL1 17.00 db
PL13 17.00 db
SFO2 400.0136001 MHz

F2 - Processing parameters
SI 32768
SF 100.6261701 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

ID NMR list parameters
EX 30.00 cm
CY 30.00 cm
F1P 220.000 MHz
F1 220.212 MHz
F2 100.626 MHz
F2P 100.626 MHz
NUC1 13C
NUC2 1H

```

100



```

Current Data Parameters
NAME      tekahshiAKM017
EXPNO     1
PROCNO    1

Date_ Acquisition Parameters
File       20090212
INSTRUM    spect
PROBHD      5 mm QNP 1H/13
PULPROG     zgpg30
TD          65536
AQ          0.033
SOLVENT     DMSO
NS          64
DS          4
SWH          6278.145 MHz
FIDRES      0.128314 Hz
AQRES       3.8664400 sec
RG           320
DE          60.400 usec
TE          6.00 usec
TE2         300.2 K
M1          1.00000000 sec
M2          0.00000000 sec
M3          0.01500000 sec

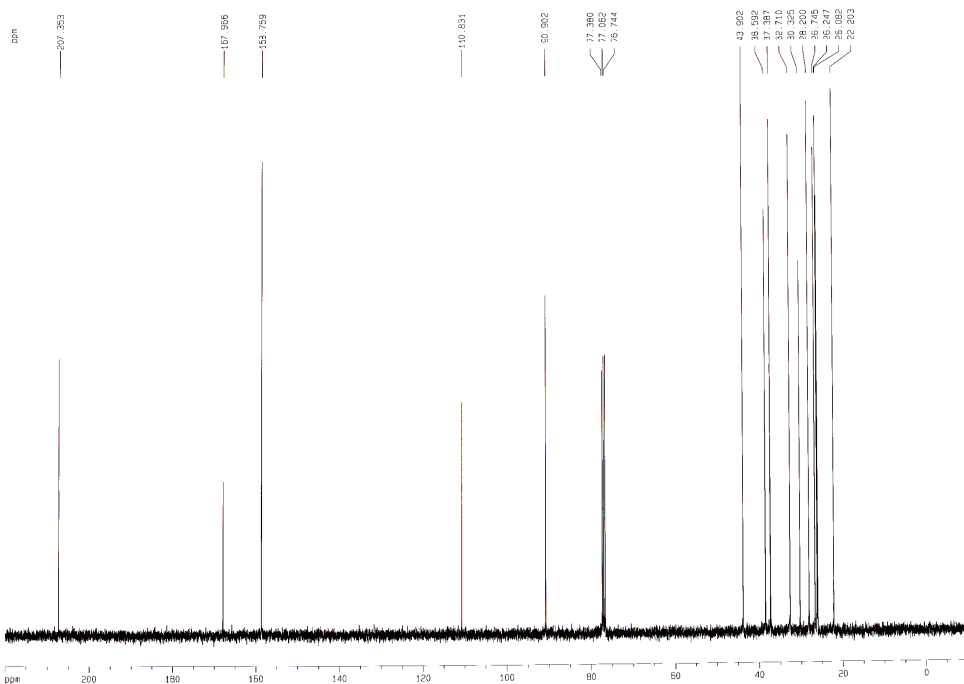
***** CHANNEL f1 *****
NUC1        13C
P1          11.50 usec
PL1         -4.00 dB
SFO1        400.6254703 MHz

2 - Processing parameters
NAME      276768
EXPNO     400.030000 MHz
INSTRUM    MSL
SOLVENT     DMSO
SSB         0
LB          0.30 Hz
RG          320
PC          1.00

TD MMR plot parameters
CX          32.00 cm
CY          21.00 cm
FZ          10.000 ppm
F1          4000.000 MHz
F2          0.000 ppm
F3          0.000 Hz
FWHMCH      0.3125 ppm/c
H2CUM      125.00938 Hz/cm

```

DPX400 carbon



```

Current Data Parameters
NAME      takahashiAM017
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20090212
Time      23.16
INSTRUM    spect
PROBHD     5 mm QNP 1H/13
PULPROG    zgpg30
TD         65536
AQ         0.0013
SOLVENT    CDCl3
NS         7
DS         4
SWH         26740.010 Hz
FIDRES     0.39945 Hz
AQRES      1,251.877 Hz
RG          1.251877
DE         10.1900 usec
TE         5.00 usec
TE2        2.000000000 sec
D11         0.00300000 sec
D12         1.000000000 sec
DEQUEST    0.00300000 sec
NMARK      0.01500000 sec

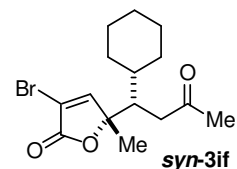
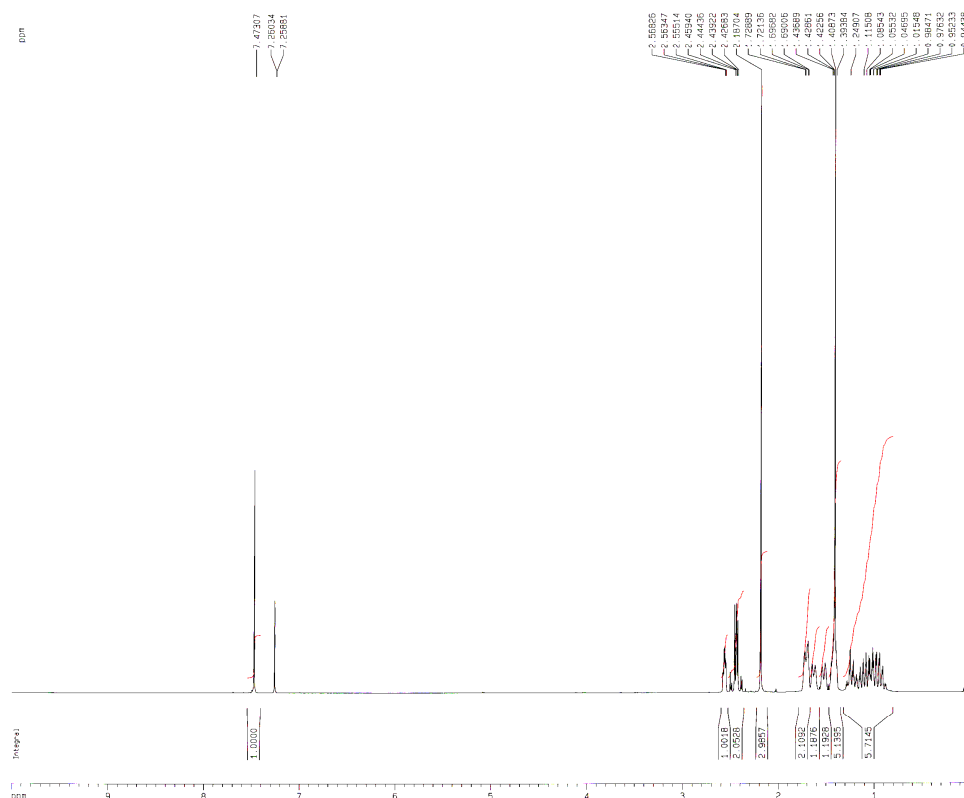
===== CHANNEL f1 =====
NUC1        13C
P1          13.00 usec
TE          5.00 usec
SFO1        100.5088060 MHz

===== CHANNEL f2 =====
CPDPRG2     null216
NUC2         1H
P2          10.00 usec
TE          5.00 usec
PL1         0.00 dB
PL2         17.00 dB
PL12         0.00 dB
PL13         17.00 dB
SFO2        400.0136100 MHz

F2 - Processing parameters
SI          32768
SF          100.5078058 MHz
WDW         EM
SSB         0
GB          0.1 Hz
PC          1.40

F2 NMR parameters
CX          32.00 cm
C1          19.00 ucm
F1          220.00 MHz
F2          221.25 MHz
F2P         -10.000 ppm
F2P2        100.628 Hz
F2P3        7.185000000 Hz
HDCM        722.9753 Hz/cm

```



```

Current Data Parameters
NAME          takahashiAMK018
EXPNO         1
PROCNO        1

Date_          Acquisition Parameters
-----
F2 -                20090912
PROBHD          5 mm QNP 1H/13
PULPROG         zgpg30
INSTRUM         spect
SOLVENT          DMSO
TD              65536
FIDRES          0.12814 Hz
AQ              3.568400 sec
RG              6278.146 Hz
DE              60.000 usec
DM              5.000 usec
D1              1.00000000 sec
MCREST          1
MCWRK          0.01500000 sec

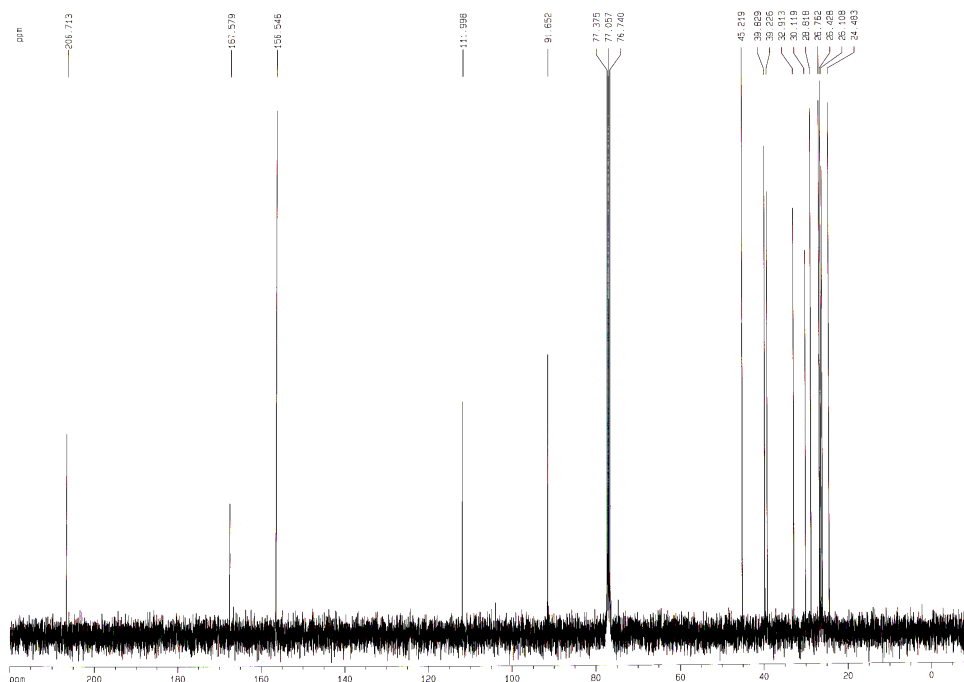
***** CHANNEL f1 *****
NUC1            13C
SF              125.761 MHz
PL1             -4.00 dB
SFO1            400.326473 MHz

F2 - Processing parameters
-----
CY              32768
SF              400.300000 MHz
SDW            0.000 MHz
SSB            0
LB              0.30 Hz
GB              0
PC              1.00

ID NMR plot parameters
-----
CX              32.00 cm
CY              21.00 cm
F1P             10.000 ppm
F2P             400.000 MHz
P1P             0.000 ppm
F2M             0.000 Hz
PCNCHN          0.314 ppm/Hz
H2ZM            125.009086 Hz/cm

```

DPX400 carbon



```

Current Data Parameters
NAME      takahashiA01018
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20090912
Time      11:30
INSTRUM    spect
PROBHD     5 mm QNP 1H/13
PULPROG    zgpg30
TD         65536
AQ         0.0313
SOLVENT     DMSO
DS          287
AS          4
SWH         26178.010 MHz
FIDRES     0.89942 Hz
AQRES      1.2517070 sec
DE         19.00 usec
TE          300.2 K
D1          2.00000000 sec
d11         0.03000000 sec
T2         1.89999999 sec
MDCRST     0.00000000 sec
MDCRST2    0.01500000 sec
MDCRST3    0.01500000 sec

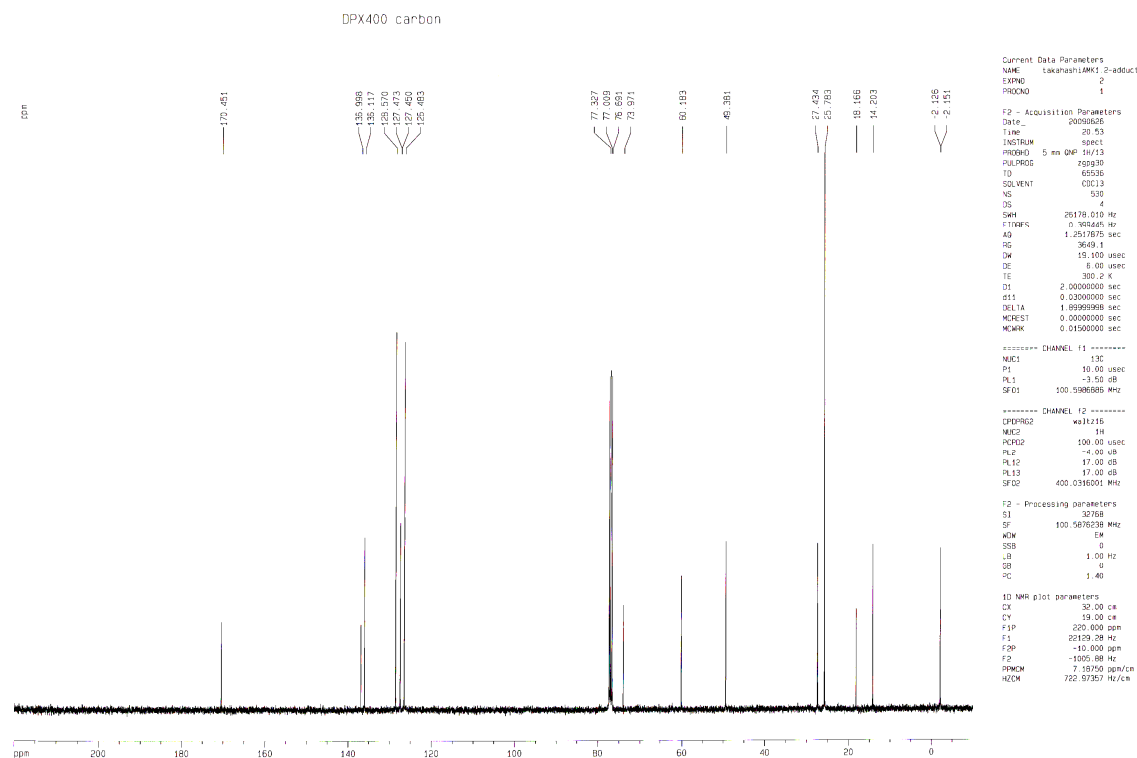
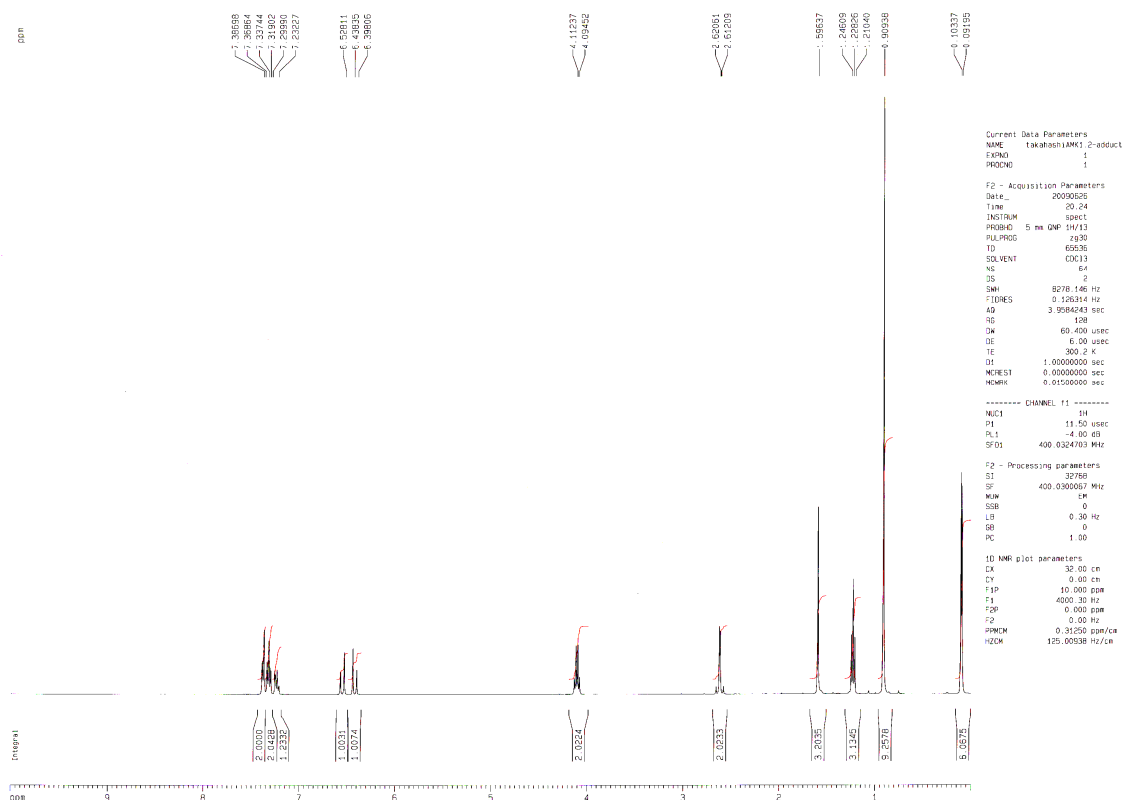
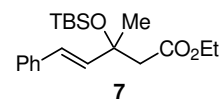
***** CHANNEL 1 *****
NUC1        13C
P1          1.00 usec
PC1         3.00 dB
SFO1        100.625806 MHz

***** CHANNEL 2 *****
CPDPRG2     waltz16
NUC2         1H
PCPD2        111 usec
PCPD22       -4.00 dB
PL12         17.00 dB
PL13         17.00 dB
SFO2        400.036100 MHz

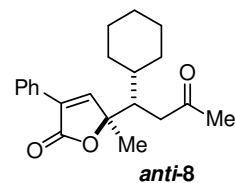
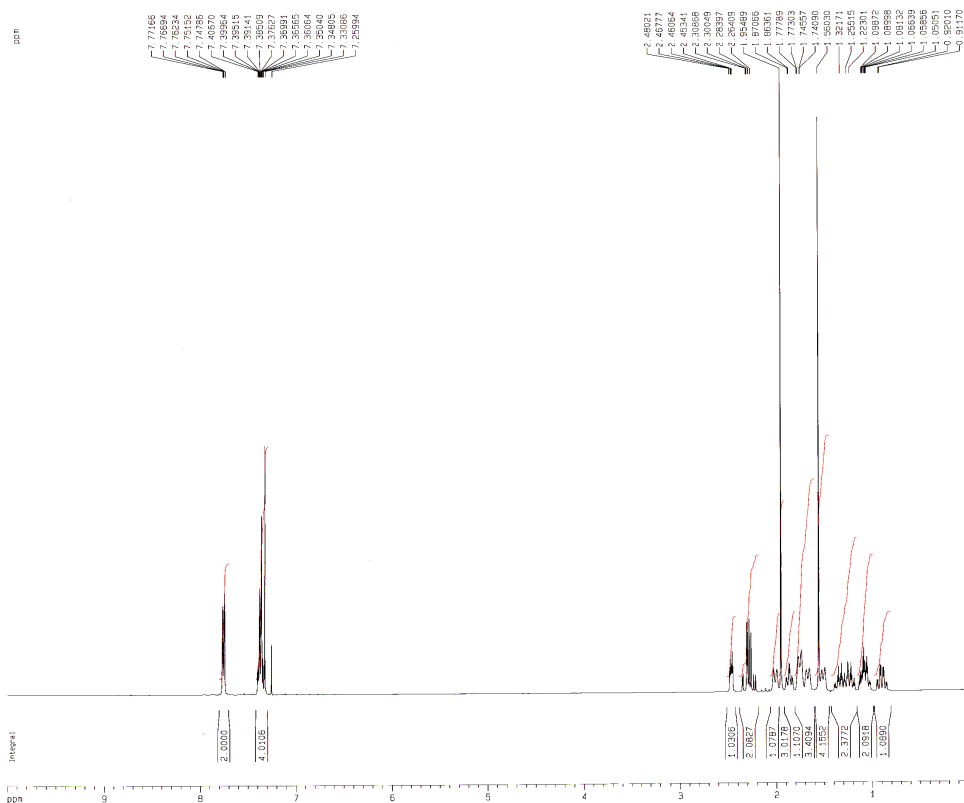
F2 - Processing parameters
SI          32768
SF           100.625806 MHz
WDW          EM
SSB          0
GB           1.00 Hz
PC           1.40

1D NMR plot parameters
X1           30.00 cm
Y1           200.00 Hz
F2           20129.20 Hz
F1           -10.000 ppm
F2           -1005.88 Hz
F1           7.10254 ppm
F2           722.95157 Hz/cm

```



DPX400 1H/13C



Current Data Parameters
NAME TAKAHASHIANK017-2
EXPNO 1
PROCNO 1

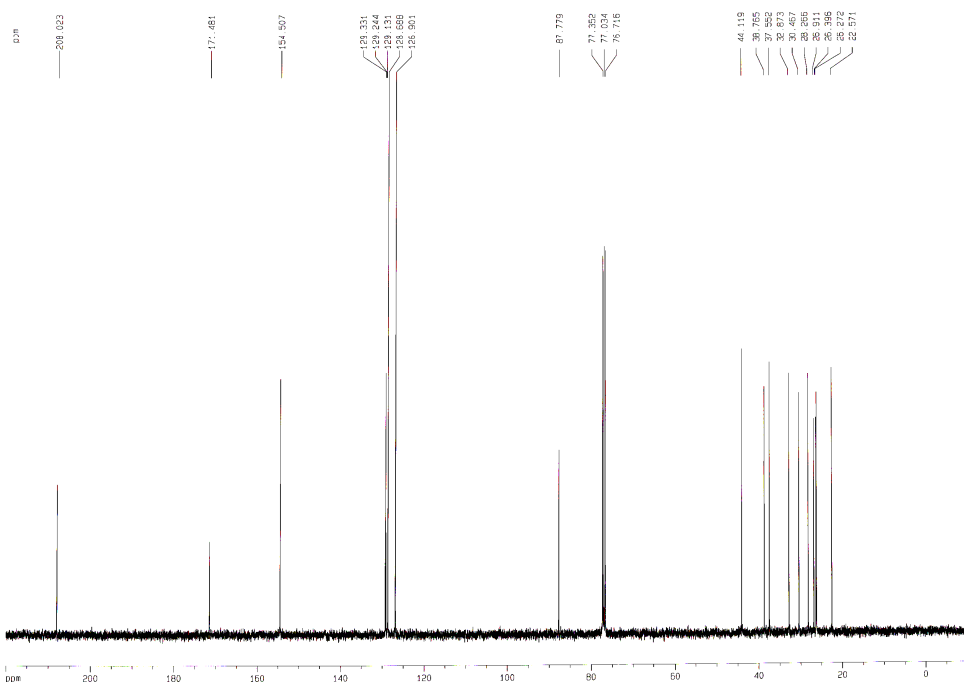
F2 - Acquisition Parameters
Date_ 20090425
Time 11.31
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 143.7
DW 50.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MDELTA 0.00000000 sec
MDELTA 0.01500000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.030055 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 21.00 cm
F1P 10.000 ppm
F1 4000.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PRNCH 0.31250 ppm/cm
HZCM 125.00938 Hz/cm

DPX400 carbon



Current Data Parameters
NAME TAKAHASHIANK017-2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090425
Time 11.35
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 223
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 4095
DW 19.100 usec
DE 5.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999999 sec
MDELTA 0.00000000 sec
MDELTA 0.01500000 sec

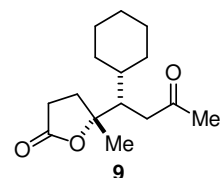
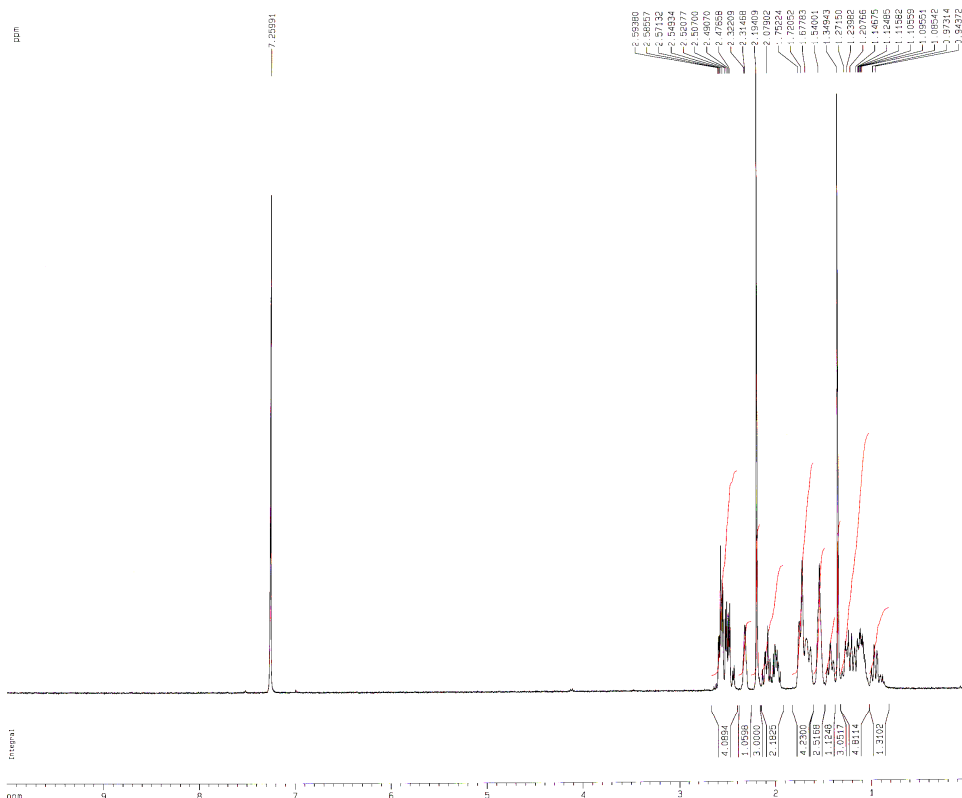
----- CHANNEL f1 -----
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986896 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876340 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 19.00 cm
F1P 220.000 ppm
F1 22129.28 Hz
F2P -10.000 ppm
F2 -1005.88 Hz
PRNCH 7.18750 ppm/cm
HZCM 722.97257 Hz/cm

3



```

Current Data Parameters
NAME      LakanahsAM017-3
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20090626
Time      22.26
INSTRUM    spect
PROBHD     5 mm NP1 1H/13
PULPROG    zgpg30
TD          65536
SOLVENT    DMS-D6
NS          2
DS          2
SWH         8278.16 Hz
FIDRES     0.1234 Hz
AQ          3.9564600 sec
RG          628.14
JE          60.400 usec
DE          6.00 usec
TE          300.2 K
MCH1ST     1.00000000 sec
MCHEST     0.00000000 sec
MCWPRG     0.01500000 sec

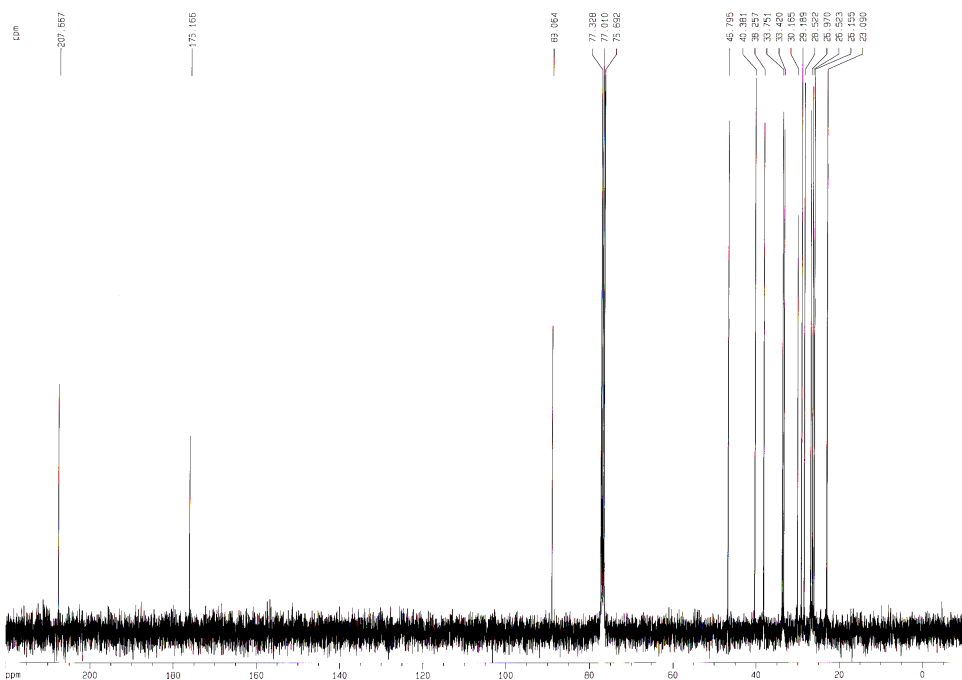
===== CHANNEL f1 =====
NUC1        13
P1          11.50 usec
PL1         -4.00 dB
SFO1        400.6234703 MHz

F2 - Processing parameters
SI          32768
SF          400.6234703 MHz
WDW          EM
SSB          0
LSD         0.30 Hz
GB          0
PC          1.00

Use non-pilot parameters
CX          31.00 cm
CY          21.00 cm
CZ          10.000 ppm
F1          400.6111111 Hz
F2          100.6200000 ppm
F3          0.00 Hz
FREQW      0.316111111 Hz
H2O         125.000000 Hz/cn

```

DPX400 carbon



```

Current Data Parameters
NAME      Lakashik/AM017-3
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_      20080826
Time       22:54
INSTRUM    smect1
PROBHD     5 mm NP14/13
PULPROG    zgpg30
RG          0.999/3.0
AQ          0.0536
TO          6.00
SOLVENT    D2O
NS          267
DS          4
SWH         26178.010 MHz
FIDRES     0.399445 Hz
AQRES      1.2517778 Hz SEC
DE          7.928 Hz
DM          6.00 usec
TE          300.2 K
D1          2.00000000 sec
D11         0.03000000 sec
DELTA       1.99999999 sec
MDCREST    0.05000000 sec
CHECK      0.01500000 sec

***** CHANNEL 1 *****
NUC1        13C
P1          10.00000000 usec
PC1         300.2 K
SFO1        100.5998880 MHz

***** CHANNEL 2 *****
PQPRG2      waltz16
MUC2        1H
PQPR2       10.00 usec
PC2         400.1460000 MHz
PL12        17.00 dB
PL13        17.00 dB
SFO2        400.0135001 MHz

F2 - Processing parameters
SI          32768
SF          100.5998880 MHz
WDW          EM
SSB          0
GB          1.00 Hz
PC          1.40

1D NMR plot parameters
CX          32.00 cm
CY          19.00 cm
F2          221.25 Hz
F1          120.00 ppm
F2P         -10.000 ppm
F1P         -7.1905888 ppm
ZC          7.1918000 ppm
ZCZ         7.1918000 ppm

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