Intermolecular Radical Reaction of O,Se-Acetals Generated via Seleno-Pummerer Rearrangement

Daisuke Urabe, Hiroki Yamaguchi, Ayumi Someya, and Masayuki Inoue*

Graduate School of Pharmaceutical Sciences
The University of Tokyo, Hongo, Bunkyo-ku, Tokyo 113-0033, Japan
Fax: (+81)3-5841-0568
E-mail: inoue@mol.f.u-tokyo.ac.jp

Supporting Information
61 page

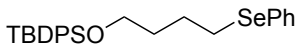
Contents: page

1. General Methods S2
2. Synthesis of selenides S2
3. Seleno-Pummerer reactions in Scheme 2 and Table 1 S4
4. Synthesis of **14d** for radical reaction S9
5. Radical reactions in Table 2 S10
6. Three-component radical reactions in Table 3 S17
7. Construction of bicyclo[5.3.0]decene structure of zedoarondiol S24
8. References S25
9. ^{1}H and ^{13}C NMR spectra of newly synthesized compounds S27

1. General methods

All reactions sensitive to air or moisture were carried out under argon atmosphere in dry or freshly distilled solvents under anhydrous conditions, unless otherwise noted. *m*-CPBA was purified by washing with pH 7 phosphate buffer and dried in vacuo (95% purity). All other reagents were used as supplied. Analytical thin-layer chromatography (TLC) was performed using E. Merck Silica gel 60 F254 pre-coated plates. Flash chromatography was performed using 50-60 μm Silica Gel 60 (Kanto Chemical Co., Inc.). Infrared (IR) spectra were recorded on JASCO FT/IR-4100 spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on JEOL JNM-ECX-500, JNM-ECA-500, or JNM-ECS-400 spectrometer. Chemical shifts were reported in ppm on the δ scale relative to $CHCl_3$ (δ = 7.26 for ^{1}H NMR), $CDCl_3$ (δ = 77.0 for ^{13}C NMR), C_6D_5H (δ = 7.16 for ^{1}H NMR), and C_6D_6 (δ = 128.0 for ^{13}C NMR) as internal references. Signal patterns are indicated as s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. High resolution mass spectra were measured on Bruker microTOFII. Elemental analysis was performed by the Analytical Laboratory at the Graduate School of Pharmaceutical Sciences, The University of Tokyo.

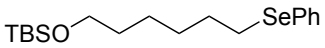
2. Syntheses of selenides



5

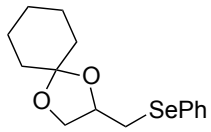
General procedure A: **Selenide 5**. MsCl (340 μL, 4.34 mmol) was added to a solution of 4-[(*tert*-butyldiphenylsilyl)oxy]butanol[1] (1.19 g, 3.62 mmol) and Et_3N (600 μL, 4.34 mmol) in THF (10 mL) at 0 °C. The reaction mixture was stirred at room temperature for 30 min, and then $(PhSe)_2$ (736 mg, 2.36 mmol), $NaBH_4$ (219 mg, 5.62 mmol) and MeOH (2 mL) were added successively. The reaction mixture was stirred for 4 h, and $NaBH_4$ (109 mg, 2.79 mmol) were added. After further 1 h, 3 N NaOH solution (3 mL) was added to the mixture. The resulting mixture was extracted with EtOAc (10 mL x 3). The combined organic layers were washed with brine, dried over Na_2SO_4, filtered and concentrated. The residue was purified by flash chromatography (silica gel 40 g, hexane/AcOEt 1:99 to 1:9) to give **5** (1.40 g, 2.99 mmol) in 83% yield: colorless oil; IR (neat) ν_{max} 2930, 2856, 1474, 1427, 1109 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.87 (9H, s, *t*-Bu of TBDPS), 1.50 (2H, tt, *J* = 6.8, 6.8 Hz, TBDPSOCH_2CH_2), 1.64 (2H, tt, *J* =7.3, 7.3 Hz, CH_2CH_2SePh), 2.73 (2H, t, *J* = 7.3 Hz, CH_2SePh), 3.49 (2H, t, *J* = 6.4 Hz, TBDPSOCH_2), 7.05-7.21 (3H, m, aromatic), 7.36-7.68 (8H, m, aromatic),

7.41-7.50 (4H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.2, 26.5, 26.8, 27.7, 32.6, 63.2, 126.6, 127.6, 129.0, 130.4, 129.6, 132.5, 133.9, 135.5; HRMS (ESI) calcd for $C_{26}H_{32}OSeSiNa$ 491.1285 ($M+Na^+$), found 491.1294.



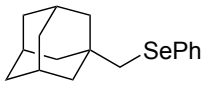
12a

Selenide 12a. According to the general procedure A, **12a** (1.27 g, 3.42 mmol) was synthesized from the known corresponding alcohol[2] (930 mg, 4.01 mmol) in 85% yield by using MsCl (470 μL, 6.03 mmol), Et_3N (1.7 mL, 12 mmol), $(PhSe)_2$ (763 mg, 2.41 mmol), $NaBH_4$ (152 mg, 4.02 mmol) and MeOH (4 mL) in THF (10 mL). The crude was purified by flash chromatography (silica gel 45 g, hexane/EtOAc 20;1): colorless oil; IR (neat) ν_{max} 2930, 2856, 1473, 1102 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.04 (6H, s, CH_3 of TBS x 2), 0.89 (9H, s, *t*-Bu of TBS), 1.31-1.54 (6H, m, CH_2 x 3), 1.71 (2H, tt, J = 7.8 Hz, $CH_2CH_2CH_2$), 2.91 (2H, t, J = 7.3 Hz, CH_2SePh), 3.58 (2H, t, J = 6.4 Hz, TBSOCH_2), 7.22-7.28 (3H, m, aromatic), 7.48 (2H, dd, J = 7.5, 1.8 Hz, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ -5.3, 18.3, 25.3, 25.9, 27.8, 29.6, 30.1, 32.6, 63.0, 126.5, 128.9, 130.6, 132.3; HRMS (ESI) calcd for $C_{18}H_{32}OSeSiNa$ 395.1285 ($M+Na^+$), found 395.1279.



12e

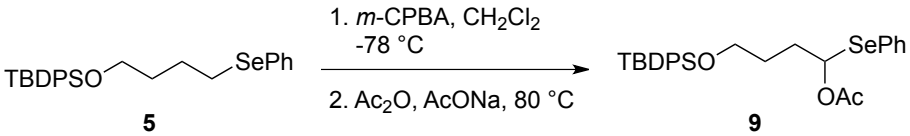
Selenide 12e. According to the general procedure A, **12e** (812 mg, 2.60 mmol) was synthesized from the known corresponding alcohol[3] (517 mg, 3.01 mmol) in 86% yield by using MsCl (350 μL, 4.47 mmol), Et_3N (630 μL, 4.55 mmol), $(PhSe)_2$ (1.15 g, 3.69 mmol), $NaBH_4$ (681 mg, 17.5 mmol) and MeOH (1.5 mL) in THF (6 mL). The crude was purified by flash chromatography (silica gel 20 g, hexane/EtOAc 1:0 to 10:1): colorless oil; IR (neat) ν_{max} 2935, 2859, 1477, 1100, 737 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.30-1.45 (2H, m, cyclopentyl), 1.49-1.64 (8H, m, cyclopentyl), 2.92 (1H, dd, J = 12.6, 8.6 Hz, CH_AH$_B$SePh), 3.18 (1H, dd, J = 12.6, 4.9 Hz, CH$_A$$H_B$SePh), 3.70 (1H, dd, J = 8.6, 6.0 Hz, OCH_AH$_B$CH), 4.08 (1H, dd, J = 8.6, 6.0 Hz, OCH$_A$$H_B$CH), 4.28 (1H, dtd, J = 8.6, 6.0, 4.9 Hz, CH_2CHCH_2), 7.25-7.27 (3H, m, aromatic), 7.51-7.53 (2H, m, aromatic); ^{13}C NMR (125 MHz, $CDCl_3$) δ 23.8, 24.0, 25.1, 30.8, 35.1, 36.7, 68.9, 75.0, 110.2, 127.2, 129.1, 129.3, 132.9; HRMS (ESI) calcd for $C_{15}H_{20}O_3SeNa$ 335.0526 ($M+Na^+$), found 335.0526.



12f

Selenide 12f. According to the general procedure A, **12f** (144 mg, 0.471 mmol) was synthesized from the commercially available corresponding alcohol (333 mg, 2.01 mmol) in 23% yield by using MsCl (460 μL, 5.87 mmol), Et_3N (840 μL, 6.07 mmol), $(PhSe)_2$ (764 mg, 2.45 mmol), $NaBH_4$ (604 mg, 15.5 mmol) and MeOH (0.4 mL) in THF (2 mL). The crude was purified by flash chromatography (silica gel 10 g, hexane/EtOAc 1:0 to 10:1): colorless oil; IR (neat) ν_{max} 2900, 2845, 1579, 1476, 1450, 1438 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.59 (6H, s, CH_2 x 3), 1.62 (3H, d, J = 12.3 Hz, CH_AH_B x 3), 1.69 (3H, d, J = 12.3 Hz, CH_AH_B x 3), 1.97 (3H, br s, CH x 3), 2.81 (2H, s, CH_2SePh), 7.20-7.26 (3H, m, aromatic), 7.50-7.52 (2H, m, aromatic); ^{13}C NMR (125 MHz, $CDCl_3$) δ 28.6, 34.0, 36.7, 42.4, 44.8, 126.3, 129.9, 132.1, 132.3; HRMS (ESI) calcd for $C_{17}H_{22}SeNa$ 329.0784 ($M+Na^+$), found 329.0786.

3. Seleno-Pummerer reactions in Scheme 2 and Table 1

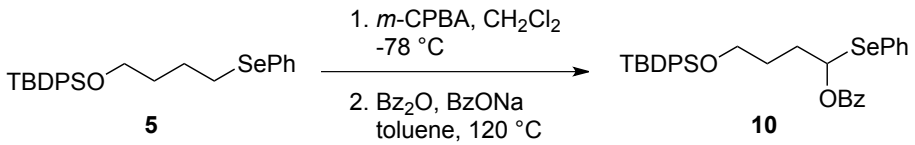


5 **9**

General procedure B: **O,Se-acetal 9**. *m*-CPBA (405 mg, 95%, 2.35 mmol) was added to a solution of selenide **5** (996 mg, 2.13 mmol) in CH_2Cl_2 (21 mL) at -78 °C. The reaction mixture was stirred at -78 °C for 30 min, and then the reaction was quenched with saturated aqueous $NaHCO_3$ (10 mL). The resultant solution was extracted with CH_2Cl_2 (10 mL x 6), and the combined organic layers were dried over Na_2SO_4 and filtered.

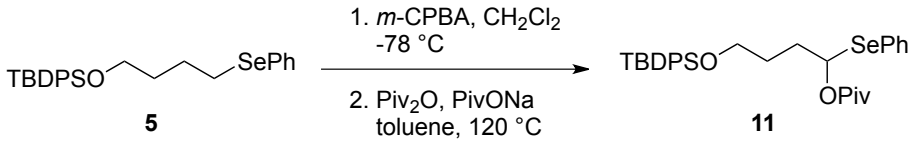
Ac_2O (21 mL) and AcONa (1.76 g, 21.5 mmol) were immediately added to the solution of the crude selenoxide. The resulting mixture was then concentrated to remove CH_2Cl_2 and the solution was heated to 80 °C for 1 h. After being cooled to room temperature, the reaction mixture was filtered and concentrated. The residue was purified by flash chromatography (silica gel 30 g, hexane/EtOAc 15:1 to 10:1) to give O,Se-acetal **9** (947 mg, 1.80 mmol) in 85% yield: pale yellow oil; IR (neat) ν_{max} 2929, 1746, 1218, 1110, 702 cm^{-1}; ^1H NMR (400 MHz, C_6D_6) δ 1.12 (9H, s, *t*-Bu of TBDPS), 1.54-1.59 (2H, m, $TBDPSOCH_2CH_2$), 1.59 (3H, s, Ac), 1.95 (2H, dt, J = 7.3, 7.3 Hz,CH_2CH_2CH), 3.50 (2H, t, J = 6.2 Hz, $TBDPSOCH_2$), 6.44 (1H, t, J = 6.2 Hz,

C*H*(SePh)OAc), 6.98-7.01 (3H, m, aromatic), 7.20-7.27 (6H, m, aromatic), 7.65-7.68 (2H, m, aromatic), 7.70-7.74 (4H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.2, 21.2, 26.8, 29.4, 32.3, 62.9, 75.1, 126.7, 127.62, 127.67, 127.68, 128.4, 128.9, 129.6, 133.71, 133.72, 135.3, 135.5, 136.1, 169.7; HRMS (ESI) calcd for $C_{28}H_{34}O_3SeSiNa$ 549.1340 ($M+Na^+$), found 549.1350.

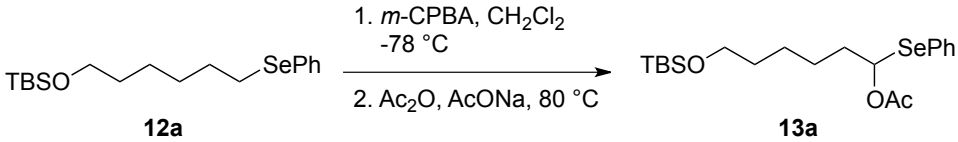


O,Se-acetal 10. *m*-CPBA (95 mg, 95%, 0.525 mmol) was added to a solution of selenide **5** (234 mg, 0.500 mmol) in CH_2Cl_2 (5 mL) at -78 °C. The reaction mixture was stirred at -78 °C for 30 min, and then the reaction was quenched with saturated aqueous $NaHCO_3$ (2 mL). The resultant solution was extracted with CH_2Cl_2 (2 mL x 6), and the combined organic layers were dried over Na_2SO_4 and filtered.

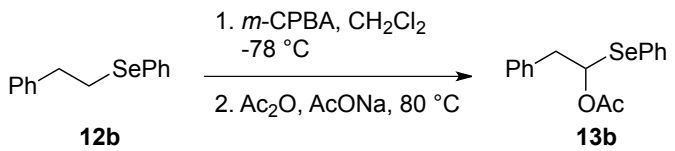
Bz_2O (566 mg, 2.50 mmol), BzONa (721 mg, 5.01 mmol), and toluene (5 mL) were immediately added to the solution of the crude selenoxide. The resulting mixture was then concentrated to remove CH_2Cl_2 and the solution was heated to 120 °C for 2h. After being cooled to room temperature, the reaction mixture was treated with H_2O (5 mL). The resulting mixture was extracted with EtOAc (5 mL x 3). The combined organic layers were dried over Na_2SO_4, filtered, and concentrated. The residue was purified by flash chromatography (silica gel 15 g, hexane/AcOEt 100:1 to 10:1) to give O,Se-acetal **10** (252 mg, 0.429 mmol) in 86% yield: colorless oil; IR (neat) ν_{max} 2955, 2930, 2857, 1723, 1428, 1264, 1110 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.01 (9H, s, *t*-Bu of TBDPS), 1.65-1.75 (2H, m, $TBDPSOCH_2CH_2$), 2.05-2.11 (2H, m, CH_2CH_2CH), 3.64 (2H, t, *J* = 6.2 Hz, $TBDPSOCH_2$), 6.46 (1H, t, *J* = 7.1 Hz, C*H*(SePh)OBz), 7.24-7.44 (12H, m, aromatic), 7.60-7.62 (6H, m, aromatic), 8.02 (2H, d, *J* = 6.8 Hz, aromatic),; ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.2, 26.8, 29.5, 32.5, 62.9, 75.6, 127.6, 128.3, 128.40, 128.43, 128.9, 129.6, 129.7, 129.9, 133.2, 133.7, 135.5, 136.4, 165.2; HRMS (ESI) calcd for $C_{33}H_{36}O_3SeSiNa$ 611.1497 ($M+Na^+$), found 611.1502.



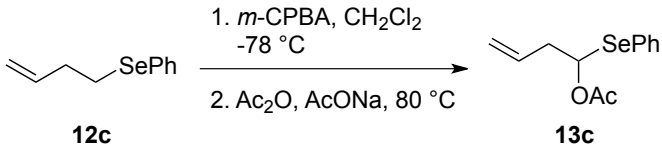
O,Se-acetal 11. According to the synthesis of **10**, O,Se-acetal **11** (242 mg, 0.426 mmol) was synthesized from selenide **5** (234 mg, 0.500 mmol) in 85% yield by using *m*-CPBA (95 mg, 95%, 0.25 mmol) in CH_2Cl_2 (5 mL) for the first step, Piv_2O (500 μL, 2.47 mmol) and PivONa (621 mg, 5.01 mmol) in toluene (5 mL) for the second step. The crude was purified by flash chromatography (silica gel 15 g, hexane/EtOAc 100:1 to 10:1): colorless oil; IR (neat) ν_{max} 2958, 2931, 1734, 1476, 1137, 1111 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.02 (9H, s, *t*-Bu of TBDPS), 1.17 (9H, s, *t*-Bu of Piv), 1.61-1.67 (2H, m, $TBDPSOCH_2CH_2$), 1.92-2.00 (2H, m, CH_2CH_2CH), 3.62 (2H, t, *J* = 6.3 Hz, $TBDPSOCH_2$), 6.24 (1H, dd, *J* = 7.8, 5.8 Hz, C*H*(SePh)OPiv), 7.25-7.44 (9H, m, aromatic), 7.59-7.63 (6H, m, aromatic); ^{13}C NMR (125 MHz, $CDCl_3$) δ 19.2, 26.8, 26.9, 29.5, 32.4, 38.8, 62.9, 74.9, 126.8, 127.6, 128.3, 128.9, 129.6, 133.8, 135.5, 136.0, 177.0; HRMS (ESI) calcd for $C_{31}H_{40}O_3SeSiNa$ 591.1810 ($M+Na^+$), found 591.1819.



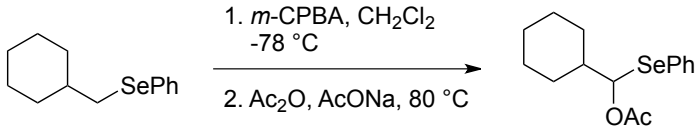
O,Se-acetal 13a. According to the general procedure B, O,Se-acetal **13a** (159 mg, 0.37 mmol) was synthesized from selenide **12a** (185 mg, 0.50 mmol) in 74% yield by using *m*-CPBA (95 mg, 95%, 0.55 mmol) in CH_2Cl_2 (5 mL) for the first step, Ac_2O (5 mL) and AcONa (409 mg, 5.00 mmol) for the second step. The crude was purified by flash chromatography (silica gel 15 g, hexane/EtOAc 20:1): colorless oil; IR (neat) ν_{max} 2931, 2857, 1748, 1221 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.03 (6H, s, CH_3 of TBS x 2), 0.88 (9H, s, *t*-Bu of TBS), 1.24-1.53 (6H, m, CH_2 x 3), 1.83 (2H, dt, *J* = 6.9 Hz, CH_2CH), 2.03 (3H, s, Ac), 3.57 (2H, t, *J* = 6.4 Hz, $TBSOCH_2$), 6.23 (1H, t, *J* = 6.9 Hz, C*H*(SePh)OAc), 7.26-7.34 (3H, m, aromatic), 7.60 (2H, dd, *J* = 8.2, 1.4 Hz, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ -5.3, 18.3, 21.1, 25.2, 25.9, 26.2, 32.6, 35.7, 62.9, 75.4, 126.9, 128.3, 128.9, 136.0, 169.7; HRMS (ESI) calcd for $C_{20}H_{34}O_3SeSiNa$ 453.1340 ($M+Na^+$), found 453.1350.



O,Se-acetal 13b: According to the general procedure B, O,Se-acetal **13b** (131 mg, 0.409 mmol) was synthesized from the known selenide **12b**[4] (122 mg, 0.467 mmol) in 88% yield by using *m*-CPBA (103 mg, 95%, 0.69 mmol) in CH_2Cl_2 (5 mL) for the first step, Ac_2O (5 mL) and AcONa (443 mg, 5.4 mmol) for the second step. The crude was purified by flash chromatography (silica gel 5 g, hexane/EtOAc 50:1 to 10:1): colorless oil; IR (neat) ν_{max} 1746, 1369, 1217, 1017 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.97 (3H, s, Ac), 3.13 (1H, dd, J = 14.3, 8.1 Hz, PhCH_AH$_B$), 3.19 (1H, dd, J = 14.3, 5.6 Hz, PhCH$_A$$H_B$), 6.42 (1H, dd, J = 8.1, 5.6 Hz, CH(SePh)OAc), 7.17 (2H, dd, J = 8.3, 1.5 Hz, aromatic), 7.23-7.36 (6H, m, aromatic), 7.59-7.60 (2H, m, aromatic); ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.0, 42.2, 75.0, 126.89, 126.91, 128.4, 128.5, 129.0, 129.3, 136.1, 136.9, 169.4; HRMS (ESI) calcd for $C_{16}H_{16}O_2Se$ 343.0213 ($M+Na^+$), found 343.0213.

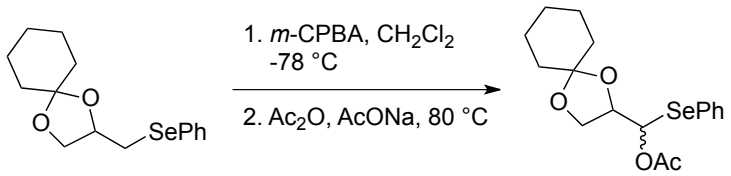


O,Se-acetal 13c. According to the general procedure B, O,Se-acetal **13c** (1.09 g, 4.04 mmol) was synthesized from the known selenide **12c**[5] (1.79 g, 8.44 mmol) in 48% yield by using *m*-CPBA (1.62 g, 95%, 9.42 mmol) in CH_2Cl_2 (42 mL) for the first step, Ac_2O (42 mL) and AcONa (6.91 g, 84.3mmol) for the second step. The crude was purified by flash chromatography (silica gel 75 g, hexane/EtOAc 30:1): colorless oil; IR (neat) ν_{max} 1746, 1438, 1370, 1218, 1017 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.03 (3H, s, OAc), 2.57-2.64 (2H, m, CH_2), 5.10 (1H, d, J = 17.5 Hz, CH_AH$_B$=CH), 5.11 (1H, d, J = 10.3 Hz, CH$_A$$H_B$=CH), 5.76 (1H, ddt, J = 17.5, 10.3, 7.3 Hz, CH$_2$=CH), 6.27 (1H, t, J = 7.3 Hz, CH(OAc)SePh), 7.27-7.37 (3H, m , aromatic), 7.59-7.63 (2H, m, aromatic); ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.1, 40.0, 74.0, 118.4, 126.7, 128.4, 129.0, 132.9, 136.0, 169.6; Anal. Calcd for $C_{12}H_{14}O_2Se$: C 53.54, H 5.24, N 0.00. Found: C 53.55, H 5.15, N 0.00.



12d **13d**

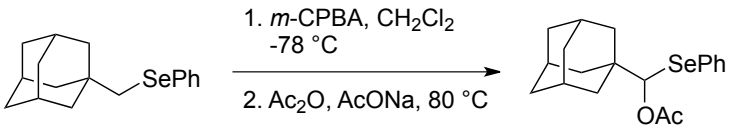
O,Se-acetal 13d. According to the general procedure B, O,Se-acetal **13d** (122 mg, 0.392 mmol) was synthesized from the known selenide **12d**[6] (127 mg, 0.500 mmol) in 78% yield by using *m*-CPBA (96 mg, 95%, 0.53 mmol) in CH_2Cl_2 (5 mL) for the first step, Ac_2O (5 mL) and AcONa (410 mg, 5.00 mmol) for the second step. The crude was purified by flash chromatography (silica gel 5 g, hexane/EtOAc 100:1 to 20:1): colorless oil; IR (neat) ν_{max} 2927, 2852, 1749, 1220, 1017 cm^{-1}; ^1H NMR (400 MHz, C_6D_6,) δ 0.93-1.11 (6H, m, CH_AH$_B$ of cyclohexyl x 6), 1.42-1.61 (3H, m. CH$_A$$H_B$ of cyclohexyl x 3), 1.61 (3H, s Ac), 1.70-1.82 (2H, m, CH$_A$$H_B$ of cyclohexyl x 2), 1.92 (1H, m, C*H*CHOAc), 6.41 (1H, d, *J* = 6.4 Hz, C*H*(SePh)OAc), 6.98-7.02 (3H, m, aromatic), 7.72-7.75 (2H, m, aromatic); ^{13}C NMR (100 MHz, C_6D_6) δ 20.5, 25.9, 26.0, 26.4, 29.4, 30.1, 43.6, 81.9, 128.5, 128.7, 129.2, 135.9, 168.9; HRMS (ESI) calcd for $C_{15}H_{20}O_2SeNa$ 335.0526 ($M+Na^+$), found 335.0523.



12e **13e**

O,Se-acetal 13e: According to the general procedure B, O,Se-acetal **13e** (170 mg, 0.459 mmol) was synthesized from selenide **12e** (156 mg, 0.502 mmol) in 92% yield as an inseparable 1:1 epimeric mixture at the acetoxy-substituted carbon center by using *m*-CPBA (95 mg, 95%, 0.52 mmol) in CH_2Cl_2 (5 mL) for the first step, Ac_2O (5 mL) and AcONa (410 mg, 5.00 mmol) for the second step. The crude was purified by flash chromatography (silica gel 5 g, hexane/EtOAc 100:1 to 10:1): colorless oil; IR (neat) ν_{max} 2936, 1751, 1367, 1217, 1021 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.30-1.48 (4H x 1/2, m, cyclopentyl), 1.50-1.63 (16H x 1/2, m, cyclopentyl), 2.06 (3H x 1/2, s, Ac), 2.12 (3H x 1/2, s, Ac), 3.86 (1H x 1/2, dd, *J* = 8.7, 6.5 Hz, OCH_AH$_B$CHO), 3.92 (1H x 1/2, dd, *J* = 8.7, 6.5 Hz, OCH_AH$_B$CHO), 4.01 (1H x 1/2, dd, *J* = 8.7, 6.5 Hz, OCH$_A$$H_B$CHO), 4.09 (1H x 1/2, dd, *J* = 8.7, 6.5 Hz, OCH$_A$$H_B$CHO), 4.27 (1H x 1/2, ddd, *J* = 8.7, 6.5, 6.5 Hz, CH$_2$C*H*CH), 4.36 (1H x 1/2, ddd, *J* = 6.5, 6.5, 4.2 Hz, CH$_2$C*H*CH), 6.11 (1H x 1/2, d, *J* = 8.7 Hz, C*H*(SePh)OAc), 6.30 (1H x 1/2, d, *J* = 4.2 Hz, C*H*(SePh)OAc), 7.25-7.36 (6H, m, aromatic), 7.60-7.65 (4H, m, aromatic); ^{13}C NMR (125 MHz, $CDCl_3$)

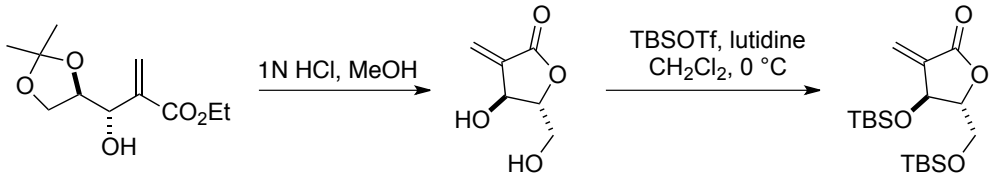
δ 21.0, 21.2, 23.71, 23.71, 23.83, 23.85, 24.98, 25.03, 34.8, 35.2, 35.8, 36.1, 65.6, 66.5, 75.6, 76.0, 76.5, 76.9, 110.8, 111.3, 125.7, 126.6, 128.6, 128.7, 129.1, 129.2, 135.9, 136.0, 169.3, 169.5; HRMS (ESI) calcd for $C_{17}H_{22}O_4SeNa$ 393.0581 ($M+Na^+$), found 393.0597.



12f **13f**

O,Se-acetal 12f. According to the general procedure B, O,Se-acetal **13f** (153 mg, 0.420 mmol) was synthesized from selenide **12f** (153 mg, 0.500 mmol) in 84% yield by using *m*-CPBA (96 mg, 95%, 0.55 mmol) in CH_2Cl_2 (5 mL) for the first step, Ac_2O (5 mL) and AcONa (410 mg, 5.00 mmol) for the second step. The crude was purified by flash chromatography (silica gel 5 g, hexane/EtOAc 100:1 to 20:1): colorless oil; IR (neat) ν_{max} 2903, 2848, 1749, 1220, 1017 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.62 (3H, d, J = 10.9 Hz, CH_AH_B x 3), 1.64 (3H, d, J = 12.6 Hz, CH_AH_B x 3), 1.71 (3H, d, J = 10.9 Hz, CH_AH_B x 3), 1.74 (3H, d, J = 12.6 Hz, CH_AH_B x 3), 6.07 (1H, s, C*H*(SePh)OAc), 7.24-7.30 (3H, m, aromatic), 7.64 (2H, dd, J = 8.6, 1.7 Hz, aromatic); ^{13}C NMR (125 MHz, $CDCl_3$) δ 20.8, 28.2, 36.7, 38.6, 38.7, 87.7, 128.0, 128.4, 128.9, 135.7, 169.7; HRMS (ESI) calcd for $C_{19}H_{24}O_2SeNa$ 387.0839 ($M+Na^+$), found 387.0831.

4. Synthesis of 14d for radical reactions

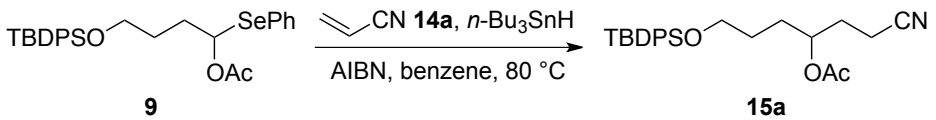


14d

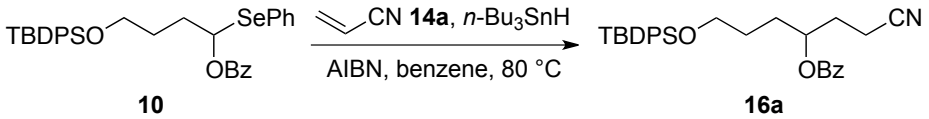
TBS ether 14d. A solution of the known allyl alcohol[7] (9.4 g, 41 mmol) in a mixture of 1N HCl (35 mL) and MeOH (230 mL) was stirred at room temperature for 12 h, and then concentrated. TBSOTf (34 mL, 150 mmol) was added to a mixture of the crude diol and 2,6-lutidine (41 mL, 350 mmol) in DMF at 0 °C. The reaction mixture was stirred at 0 °C for 3 h, and the reaction was quenched with saturated aqueous NH_4Cl. The resultant solution was extracted with Et_2O (x 3). The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated. The residue

was purified by flash chromatography (hexane/EtOAc 100:1 to 40:1) to afford **14d** (12 g, 32 mmol) in 78% yield over 2 steps: white solid; m.p. 61-62.5 °C; $[\alpha]_D^{20}$ = 17.8 (*c* 1.61 $CHCl_3$); IR (neat) ν_{max} 2951, 2929, 2857, 1759, 1117 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.04 (3H, s, C*H*$_3$ of TBS), 0.06 (3H, s, C*H*$_3$ of TBS), 0.13 (3H, s, C*H*$_3$ of TBS), 0.14 (3H, s, C*H*$_3$ of TBS), 0.85 (9H, s, *t*-Bu of TBS), 0.90 (9H, s, *t*-Bu of TBS), 3.78 (1H, dd, *J* = 11.9, 2.3 Hz, C*H*$_A$H$_B$OTBS), 3.86 (1H, dd, *J* = 11.9, 3.2 Hz, CH$_A$*H*$_B$OTBS), 4.22 (1H, ddd, *J* = 4.1, 3.2, 2.3 Hz, C*H*CH$_2$OTBS), 4.89 (1H, ddd, *J* = 4.1, 2.3, 1.8 Hz, C*H*C=CH$_2$), 5.77 (1H, d, *J* = 1.8 Hz, C=C*H*$_A$H$_B$), 6.33 (1H, d, *J* = 2.3 Hz, C=CH$_A$*H*$_B$); ^{13}C NMR (100 MHz, $CDCl_3$) δ -5.6, -5.5, -4.6, -4.4, 17.9, 18.2, 25.6, 25.7, 61.3, 69.2, 85.1, 124.3, 139.5, 168.8; HRMS (ESI) calcd for $C_{18}H_{36}O_4Si_2Na$ 395.2050 (M+Na$^+$), found 395.2048.

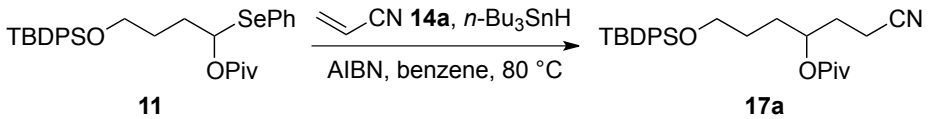
5. Radical Reaction in Table 2



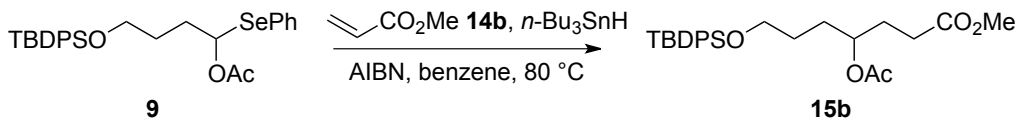
***General procedure C*: Compound 15a.** A solution of O,Se-acetal **9** (38 mg, 0.072 mmol), acrylonitrile **14a** (17 μL, 0.11 mmol) and AIBN (2.4 mg, 0.015 mmol) in benzene (1.8 mL) was degassed by freeze-thaw procedure (x 3). The mixture was heated to 80 °C, and degassed solution of *n*-Bu_3SnH (40 μL, 0.15 mmol) and AIBN (2.3 mg, 0.15 mmol) in benzene (1.8 mL) was added with a syringe pump over 1 h. After additional 1 h at 80 °C, the reaction mixture was cooled to room temperature and concentrated. The residue was purified by flash chromatography [a column consecutively packed with silica gel 15 g and 10% (w/w) KF contained silica gel 5 g, hexane/EtOAc 7:1 to 5:1] to afford **15a** (26 mg, 0.061 mmol) in 86% yield: colorless oil; IR (neat) ν_{max} 2955, 2931, 2858, 2248, 1739, 1237, 1111 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.05 (9H, s, *t*-Bu of TBDPS), 1.49-1.59 (2H, m, TBDPSOCH$_2$C*H*$_2$), 1.60-1.69 (2H, m, TBDPSOCH$_2$CH$_2$C*H*$_2$), 1.83-1.94 (2H, m, C*H*$_2$CH$_2$CN), 2.06 (3H, s, OAc), 2.34 (2H, t, *J* = 7.5 Hz, C*H*$_2$CN), 3.66 (2H, t, *J* = 6.0 Hz, TBDPSOC*H*$_2$), 4.92 (1H, m, C*H*OAc), 7.36-7.46 (6H, m, aromatic), 7.65 (4H, dd, *J* = 7.8, 1.5 Hz, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.5, 19.2, 21.0, 26.8, 28.1, 29.8, 30.2, 63.2, 72.2, 119.1, 127.7, 129.6, 133.7, 135.5, 170.7; HRMS (ESI) calcd for $C_{25}H_{33}NO_3SiNa$ 446.2127 (M+Na$^+$), found 446.2123.



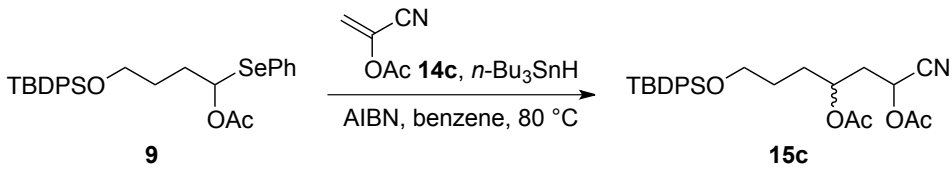
Compound 16a. According to the general procedure C, **16a** (32 mg, 0.066 mmol) was synthesized from O,Se-acetal **10** (54 mg, 0.092 mmol) in 72% yield by using acrylonitrile **14a** (9.2 μL, 0.14 mmol), *n*-Bu_3SnH (50 μL, 0.19 mmol) and AIBN (6.0 mg, 0.037 mmol) in benzene (4.6 mL). The crude was purified by flash chromatography [a column consecutively packed with silica gel 15 g and 10% (w/w) KF contained silica gel 5 g, hexane/EtOAc 20:1 to 7:1]: colorless oil; IR (neat) ν_{max} 2931, 2857, 2248, 1717, 1272, 1111 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ1.04 (9H, s, *t*-Bu of TBDPS), 1.57-1.69 (2H, m, $TBDPSOCH_2CH_2$), 1.77-1.84 (2H, m, $TBDPSOCH_2CH_2CH_2$), 2.05 (2H, td, *J* = 8.2, 6.0 Hz, CH_2CH_2CN), 2.41 (2H, t, *J* = 8.2 Hz, CH_2CN), 3.68 (td, *J* = 6.0, 1.0 Hz, $TBDPSOCH_2$), 5.19 (1H, quintet, *J* = 6.0 Hz, *C*HOBz), 7.33-7.48 (8H, m, aromatic), 7.58 (1H, tt, *J* = 7.5, 1.2 Hz, aromatic), 7.64 (4H, m, aromatic), 8.03 (2H, m, aromatic) ; ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.6, 19.2, 26.8, 28.1, 30.2, 30.3, 63.2, 72.8, 119.1, 127.6, 128.4, 129.6, 129.8, 133.2, 133.72, 133.73, 135.5, 166.1; HRMS (ESI) calcd for $C_{30}H_{35}NO_3SiNa$ 508.2284 ($M+Na^+$), found 508.2272.



Compound 17a. According to the general procedure C, **17a** (29 mg, 0.062 mmol) was synthesized from O,Se-acetal **11** (44 mg, 0.077 mmol) in 80% yield by using acrylonitrile **14a** (7.8 μL, 0.12 mmol), *n*-Bu_3SnH (42 μL, 0.16 mmol) and AIBN (5.1 mg, 0.031 mmol) in benzene (3.9 mL). The crude was purified by flash chromatography [a column consecutively packed with silica gel 15 g and 10% (w/w) KF contained silica gel 5 g, hexane/EtOAc 10:1 to 7:1]: colorless oil; IR (neat) ν_{max} 2957, 2931, 2857, 2248, 1726, 1156, 1111 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.04 (9H, s, *t*-Bu of TBDPS), 1.19 (9H, s, *t*-Bu of Piv), 1.49-1.60 (2H, m, $TBDPSOCH_2CH_2$), 1.61-1.69 (2H, m, $TBDPSOCH_2CH_2CH_2$), 1.87-1.95 (2H, m, CH_2CH_2CN), 2.31 (2H, t, *J* = 7.8 Hz, CH_2CN), 3.66 (2H, t, *J* = 6.4 Hz, $TBDPSOCH_2$), 4.89 (1H, quintet, *J* = 6.4, Hz, *C*HOPiv), 7.35-7.46 (6H, m, aromatic), 7.64 (4H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.5, 19.2, 26.8, 27.1, 28.1, 30.0, 30.2, 38.9, 63.2, 71.7, 119.1, 127.6, 129.6, 133.8, 135.5, 178.0; HRMS (ESI) calcd for $C_{28}H_{39}NO_3SiNa$ 488.2597 ($M+Na^+$), found 488.2585.

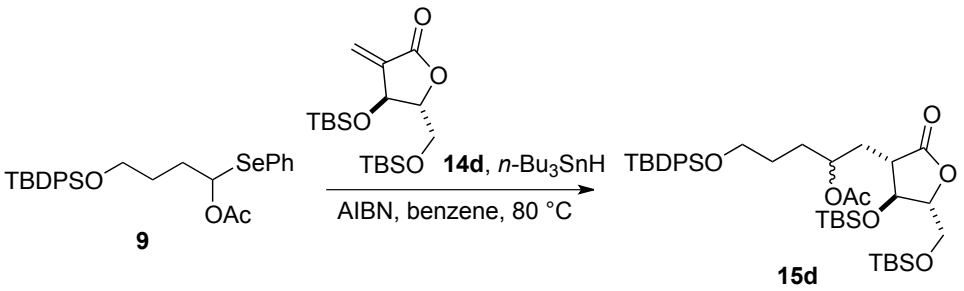


Compound 15b. According to the general procedure C, **15b** (30 mg, 0.066 mmol) was synthesized from O,Se-acetal **9** (49 mg, 0.093 mmol) in 72% yield by using methylacrylate **14b** (13 μL, 0.14 mmol), *n*-Bu_3SnH (51 μL, 0.19 mmol) and AIBN (6.1 mg, 0.037 mmol) in benzene (4.7 mL). The crude was purified by flash chromatography [a column consecutively packed with silica gel 15 g and 10% (w/w) KF contained silica gel 5 g, hexane/EtOAc 7:1 to 4:1]: colorless oil; IR (neat) ν_{max} 2953, 2858, 1739, 1240, 1111 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.04 (9H, s, *t*-Bu of TBDPS), 1.50-1.59 (2H, m, $TBDPSOCH_2CH_2$), 1.59-1.68 (2H, m, $TBDPSOCH_2CH_2CH_2$), 1.78-1.97 (2H, m, $CH_2CH_2CO_2Me$), 2.01 (3H, s, OAc), 2.32 (2H, t, *J* = 7.7 Hz, CH_2CO_2Me), 3.65 (2H, td, *J* = 6.5, 1.9 Hz, $TBDPSOCH_2$), 3.67 (3H, s, OCH_3), 4.90 (1H, m, C*H*), 7.35-7.45 (6H, m, aromatic), 7.65 (4H, dd, *J* = 8.2, 1.8 Hz, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.2, 21.1, 26.8, 28.2, 29.1, 30.0, 30.4, 51.6, 63.4, 73.0, 127.6, 129.6, 133.8, 135.5, 170.7, 173.5; HRMS (ESI) calcd for $C_{26}H_{36}O_5SiNa$ 479.2230 ($M+Na^+$), found 479.2228.



Compound 15c. According to the general procedure C, **15c** (36 mg, 0.075 mmol) was synthesized from O,Se-acetal **9** (45 mg, 0.086 mmol) in 87% yield as a 1:1 epimeric mixture at the acetoxy-substituted carbon center by using **14c** (14 μL, 0.13 mmol), *n*-Bu_3SnH (47 μL, 0.18 mmol) and AIBN (5.7 mg, 0.035 mmol) in benzene (4.3 mL). The crude was purified by flash chromatography [a column consecutively packed with silica gel 15 g and 10% (w/w) KF contained silica gel 5 g, hexane/EtOAc 7:1 to 5:1]. Analytically pure **15c polar** and **15c less polar** were obtained by separation from the epimeric mixture of **15c** using flash chromatography (silica gel 10 g, CH_2Cl_2). **15c polar**: colorless oil; IR (neat) ν_{max} 2931, 1756, 1741, 1235, 1111 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.05 (9H, s, *t*-Bu of TBDPS), 1.52-1.57 (2H, m, $TBDPSOCH_2CH_2$), 1.63-1.73 (2H, m, $TBDPSOCH_2CH_2CH_2$), 2.03 (3H, s, OAc), 2.12 (3H, s, OAc), 2.11-2.19 (2H, m $CH(OAc)CH_2CH(OAc)$), 3.65 (2H, t, *J* = 6.1 Hz, $TBDPSOCH_2$), 5.00 (1H, m, $CH_2CH(OAc)CH_2$), 5.32 (1H, dd, *J* = 9.4, 4.9 Hz, C*H*(OAc)CN), 7.38-7.43 (6H,

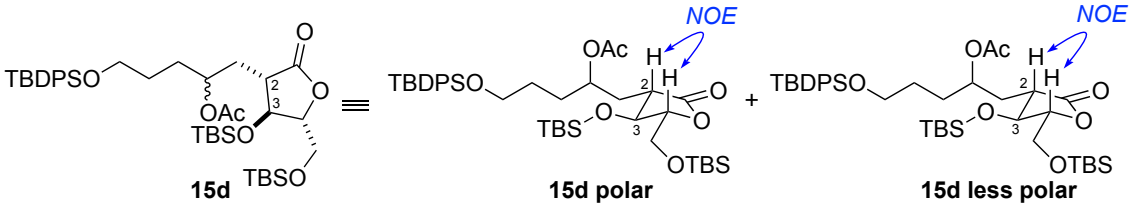
m, aromatic), 7.63-7.66 (4H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.2, 20.1, 20.9, 26.8, 27.9, 30.8, 36.9, 57.3, 63.1, 68.5, 116.7, 127.7, 129.7, 133.7, 135.5, 169.1, 170.5; HRMS (ESI) calcd for $C_{27}H_{35}NO_5SiNa$ 504.2182 (M+Na$^+$), found 504.2184. **15c less polar**: colorless oil; IR (neat) ν_{max} 2932, 1753, 1743, 1220, 1111 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.05 (9H, s, *t*-Bu of TBDPS), 1.50-1.60 (2H, m, TBDPSOCH$_2$C*H*$_2$), 1.70 (2H, q, *J* = 7.0 Hz, TBDPSOCH$_2$CH$_2$C*H*$_2$), 2.07 (3H, s, OAc), 2.13 (3H, s, OAc), 2.15 (2H, m CH(OAc)C*H*$_2$CH(OAc)), 3.66 (2H, t, *J* = 6.1 Hz, TBDPSOC*H*$_2$), 5.09 (1H, quintet, *J* = 6.1 Hz, CH$_2$C*H*(OAc)CH$_2$), 5.58 (1H, t, *J* = 6.5, Hz, C*H*(OAc)CN), 7.37-7.45 (6H, m, aromatic), 7.64-7.65 (4H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.2, 20.4, 21.0, 26.8, 27.9, 30.6, 36.4, 58.2, 63.2, 69.2, 116.4, 127.7, 129.7, 133.7, 135.5, 168.9, 170.4; HRMS (ESI) calcd for $C_{27}H_{35}NO_5SiNa$ 504.2182 (M+Na$^+$), found 504.2191.

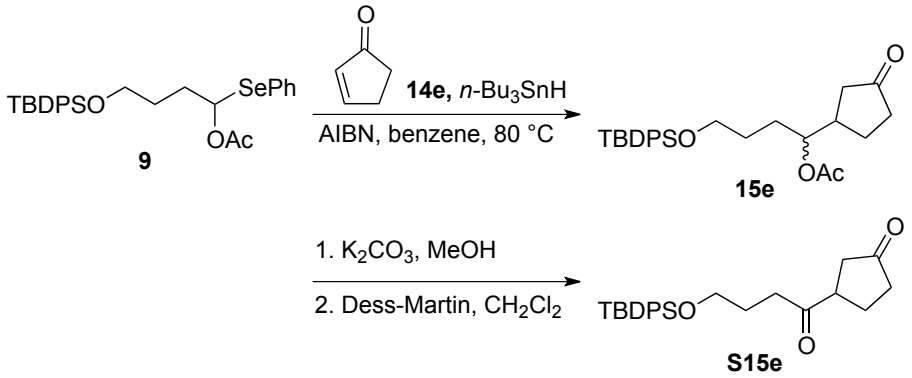


Compound 15d. According to the general procedure C, **15d** (50 mg, 0.067 mmol) was synthesized from O,Se-acetal **9** (47 mg, 0.089 mmol) in 78% yield as a 1:1 epimeric mixture at the acetoxy-substituted carbon center by using **14d** (50 mg, 0.13 mmol), *n*-Bu$_3$SnH (49 μL, 0.18 mmol) and AIBN (5.9 mg, 0.036 mmol) in benzene (4.5 mL). The crude was purified by flash chromatography [a column consecutively packed with silica gel 15 g and 10% (w/w) KF contained silica gel 5 g, hexane/EtOAc 15:1 to 10:1]. Analytically pure **15d polar** and **15d less polar** were obtained by separation from the epimeric mixture of **15d** using flash chromatography (silica gel 10 g, hexane/EtOAc 15:1). **15d polar**: colorless oil; IR (neat) ν_{max} 2953, 2929, 2857, 1786, 1739, 1252, 1112 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.06 (3H, s, C*H*$_3$ of TBS), 0.08 (3H, s, C*H*$_3$ of TBS), 0.11 (3H, s, C*H*$_3$ of TBS), 0.12 (3H, s, C*H*$_3$ of TBS), 0.89 (18H, s, *t*-Bu of TBS), 1.04 (9H, s, *t*-Bu of TBDPS), 1.50-1.77 (4H, m, TBDPSOCH$_2$C*H*$_2$C*H*$_2$), 1.83 (1H, ddd, *J* = 16.0, 8.6, 5.5 Hz, COCHC*H*$_A$H$_B$), 1.91 (1H, ddd, *J* = 16.0, 8.6, 5.5 Hz, COCHCH$_A$*H*$_B$), 2.01 (3H, s, OAc), 2.56 (1H, m, COC*H*), 3.65 (2H, t, *J* = 6.0 Hz, TBDPSOC*H*$_2$), 3.74 (1H, dd, *J* = 12.0, 3.0 Hz, TBSOC*H*$_A$H$_B$), 3.91 (1H, dd, *J* = 12.0, 2.2 Hz, TBSOCH$_A$*H*$_B$), 4.11 (1H, ddd, *J* = 5.8, 3.0, 2.2 Hz, TBSOCH$_2$C*H*), 4.28 (1H, dd,

J = 6.8, 5.8 Hz, TBSOC*H*), 5.19 (1H, m, C*H*OAc), 7.35-7.45 (6H, m, aromatic), 7.65 (4H, d, J = 7.2 Hz, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ -5.5, -5.3, -4.5, -4.4, 17.8, 18.3, 19.2, 21.1, 25.6, 25.8, 26.8, 28.1, 30.6, 33.1, 46.0, 61.0, 63.5, 71.6, 72.9, 84.9, 127.6, 129.6, 133.9, 135.5, 170.6, 175.9; HRMS (ESI) calcd for $C_{40}H_{66}O_7Si_3Na$ 765.4014 (M+Na$^+$), found 765.4003. **15d less polar**: colorless oil; IR (neat) ν_{max} 2930, 2857, 1785, 1740, 1253, 1112 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.06 (3H, s, CH_3 of TBS), 0.08 (3H, s, CH_3 of TBS), 0.09 (3H, s, CH_3 of TBS), 0.11 (3H, s, CH_3 of TBS), 0.88 (18H, s, *t*-Bu of TBS), 1.04 (9H, s, *t*-Bu of TBDPS), 1.50-1.85 (5H, m, COCHCH_AH$_B$, TBDPSOCH$_2$CH_2CH_2), 2.02 (3H, s, OAc), 2.08 (1H, ddd, J = 14.6, 8.7, 6.9 Hz, COCHCH$_A$$H_B$), 2.66 (1H, q, J = 6.9 Hz, COC*H*), 3.64 (2H, t, J = 6.0 Hz, TBDPSOCH_2), 3.76 (1H, dd, J = 11.9, 2.3 Hz, TBSOCH_AH$_B$), 3.88 (1H, dd, J = 11.9, 2.3 Hz, TBSOCH$_A$$H_B$), 4.15 (1H, dt, J = 5.0, 2.3 Hz, TBSOCH$_2$C*H*), 4.28 (1H, dd, J = 6.9, 5.0 Hz, TBSOC*H*), 5.09 (1H, m, C*H*OAc), 7.34-7.44 (6H, m, aromatic), 7.65 (4H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ -5.5, -5.3, -4.5, -4.4, 17.7, 18.4, 19.2, 21.2, 25.6, 25.9, 26.8, 28.0, 30.5, 33.1, 46.8, 61.1, 63.5, 71.9, 73.5, 85.4, 127.6, 129.6, 133.8, 135.5, 170.5, 176.3; HRMS (ESI) calcd for $C_{40}H_{66}O_7Si_3Na$ 765.4014 (M+Na$^+$), found 765.3995.

The structure of 2,3-*trans*-substituted γ-lactone of **15d** was determined by the NOE experiments after separation of the epimeric mixture of **15d**.





Compound 15e. A solution of O,Se-acetal **9** (40 mg, 0.076 mmol), **14e** (32 μL, 0.39

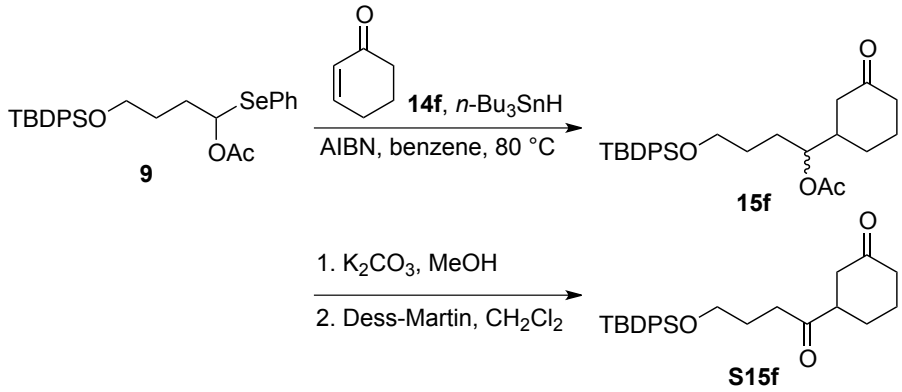
mmol) and AIBN (2.4 mg, 0.015 mmol) in benzene (1.8 mL) was degassed by freeze-thaw procedure (x 3). The mixture was heated to 80 °C, and degassed solution of *n*-Bu_3SnH (125 μL, 0.46 mmol) and AIBN (2.3 mg, 0.15 mmol) in benzene (1.8 mL) was added with a syringe pump over 3 h. After additional 1 h at 80 °C, the reaction mixture was cooled to room temperature and concentrated. The residue was purified by flash chromatography [a column consecutively packed with silica gel 15 g and 10% (w/w) KF contained silica gel 5 g, hexane/EtOAc 30:1 to 5:1] to afford **15e** (24 mg, 0.053 mmol) in 70% yield as a 1:1 epimeric mixture at the acetoxy-substituted carbon center: ^{1}H NMR (400 MHz, $CDCl_3$) of a 1:1 epimeric mixture of **15e** δ 1.04 (9H, s, *t*-Bu of TBDPS), 1.50-1.75 (5H, m), 1.85-2.42 (6H, m), 2.03 (3H x 1/2, s, Ac), 2.04 (3H x 1/2, s, Ac), 3.62-3.70 (2H, m, TBDPSOCH_2), 4.90-5.01 (1H, m, AcOC*H*), 7.35-7.46 (6H, m, aromatic), 7.65 (4H, d, *J* = 8.5 Hz, aromatic); HRMS (ESI) calcd for $C_{27}H_{36}O_4SiNa$ 475.2281 ($M+Na^+$), found 475.2288.

Since the epimeric mixture of **15e** was inseparable, the structure of the product was established from the NMR data of diketone **S15e**, which was prepared through deacetylation and subsequent oxidation.

General procedure D: **Diketone S15e.** A 1:1 epimeric mixture of **15e** (24 mg, 0.053 mmol) and K_2CO_3 (22 mg, 0.16 mmol) in MeOH (1.1 mL) was stirred for 14 h at room temperature. After addition of saturated aqueous NH_4Cl (2 mL), the mixture was extracted with CH_2Cl_2 (2 mL x 3). The combined organic layers were dried over Na_2SO_4, filtered, and concentrated to give the crude alcohol, which was used to the next reaction without further purification.

Dess-Martin periodinane (44 mg, 0.10 mmol) was added to a solution of the crude alcohol in CH_2Cl_2 (1.1 mL) at 0 °C. After the reaction mixture was stirred for 1 h at room temperature, saturated aqueous $Na_2S_2O_3$ (2 mL) was added. The mixture was extracted with CH_2Cl_2 (2 mL x 3). The combined organic layers were dried over Na_2SO_4, filtered, and concentrated. The residue was purified by flash chromatography (silica gel 5 g, hexane/EtOAc 7:1) to afford ketone **S15e** (20 mg, 0.049 mmol) in 93% yield over 2 steps: colorless oil; IR (neat) ν_{max} 2930, 2856, 1743, 1709, 1427, 1109 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.05 (9H, s, *t*-Bu of TBDPS), 1.85 (2H, quintet, *J* = 7.0 Hz, TBDPSOCH_2CH_2), 1.97 (1H, ddd, *J* = 17.0, 12.4, 8.6 Hz, $COCH_2CH_AH_B$), 2.15-2.38 (4H, m, COC$H_2$$CH_AH_BCHCH_A$$H_B$), 2.48 (1H, dd, *J* = 18.3, 7.8 Hz, $COCH_AH_BCH$), 2.59 (1H, dt, *J* = 17.9, 7.3 Hz, TBDPSO$CH_2CH_2CH_AH_B$), 2.70 (1H, dt, *J* = 17.9, 7.3 Hz, TBDPSO$CH_2CH_2CH_AH_B$), 3.21 (1H, br quintet, *J* = 7.6 Hz, $COCH_2$C*H*), 3.69 (2H, t, *J* = 6.0 Hz, TBDPSOCH_2), 7.35-7.46 (6H, m, aromatic), 7.64 (4H, dd, *J* = 7.8, 1.4 Hz, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ19.2, 26.0, 26.4, 26.8,

37.5, 37.8, 40.0, 47.8, 62.8, 127.7, 129.7, 133.7, 135.5, 210.4, 216.7; HRMS (ESI) calcd for $C_{25}H_{32}O_3SiNa$ 431.2018 ($M+Na^+$), found 431.2023.



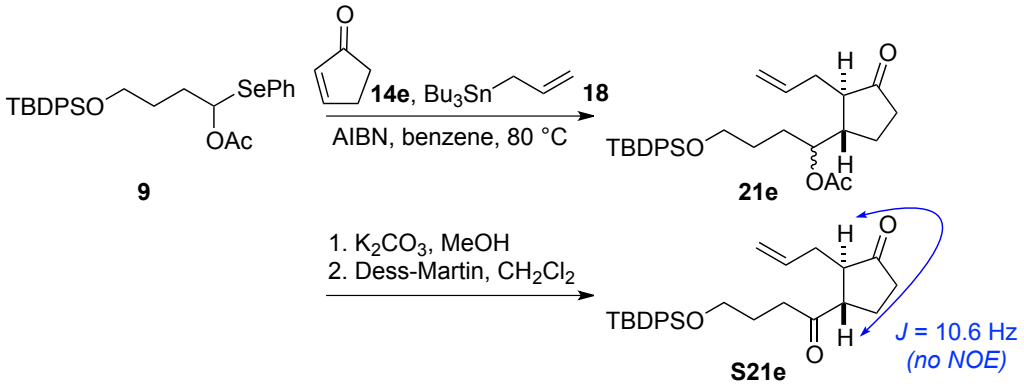
Compound 15f. A solution of O,Se-acetal **9** (53 mg, 0.10 mmol), **14f** (48 μL, 0.50 mmol) and AIBN (3.3 mg, 0.020 mmol) in benzene (2.5 mL) was degassed by freeze-thaw procedure (x 3). The mixture was heated at 80 °C, and degassed solution of *n*-Bu_3SnH (165 μL, 0.61 mmol) and AIBN (3.3 mg, 0.020 mmol) in benzene (2.5 mL) was added with a syringe pump over 3 h. After additional 1 h at 80 °C, the reaction mixture was cooled to room temperature and concentrated. The residue was purified by flash chromatography [a column consecutively packed with silica gel 15 g and 10% (w/w) KF contained silica gel 5 g, CH_2Cl_2/EtOAc 30:1] to afford **15f** (10 mg, 0.022 mmol) in 22% yield as a 3:2 epimeric mixture at the acetoxy-substituted carbon center: ^{1}H NMR of a 3:2 epimeric mixture of **15f** (400 MHz, $CDCl_3$) δ 1.04 (9H, s, *t*-Bu of TBDPS), 1.20-2.45 (26H, m), 2.03 (3H x 2/5, s, OAc), 2.04 (3H x 3/5, s, Ac), 3.60-3.71 (2H, m, TBDPSOCH_2), 4.82 (1H x 2/5, m, C*H*OAc), 4.89 (1H x 3/5, dt, J = 8.2, 5.0 Hz, C*H*OAc), 7.35-7.45 (6H, m, aromatic), 7.62-7.68 (4H, m, aromatic); LRMS (ESI) calcd for $C_{28}H_{38}O_4SiNa$ 489 ($M+Na^+$), found 489.

Since the epimeric mixture of **15f** was inseparable, the structure of the product was established from the NMR data of diketone **S15f**, which was prepared through deacetylation and subsequent oxidation.

Diketone S15f. According to the general procedure D, diketone **S15f** (2.5 mg, 5.9 μmol) was synthesized in 81% yield over 2 steps from a 3:2 epimeric mixture of **15f** (3.4 mg, 7.3 μmol) by using K_2CO_3 (3.2 mg, 0.023 mmol) in MeOH (0.15 mL) for the first step, and Dess-Martin periodinane (6.2 mg, 0.015 mmol) in CH_2Cl_2 (0.15 mL) for the second step. The crude was purified by flash chromatography (silica gel 2 g, hexane/EtOAc 7:1): colorless oil; IR (neat) ν_{max} 2931, 1712, 1427, 1111 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.05 (9H, s, *t*-Bu of TBDPS), 1.60-1.76 (2H, m,

$COCH_2CH_AH_BCH_AH_B$), 1.82 (2H, quintet, J = 6.6 Hz, $TBDPSOCH_2CH_2$), 1.99-2.12 (2H, m, $COCH_2CH_AH_BCH_AH_B$), 2.25-2.42 (3H, m, $COCH_2CH_2CH_2CHCH_AH_B$), 2.50 (1H, dd, J = 16.0, 11.0 Hz, $COCH_AH_B$), 2.52 (1H, dt, J = 17.4, 7.3 Hz, $TBDPSOCH_2CH_2CH_AH_B$), 2.65 (1H, dt, J = 17.4, 7.8 Hz, $TBDPSOCH_2CH_2CH_AH_B$), 2.84 (1H, m, COC*H*), 3.67 (2H, t, J = 5.9 Hz, $TBDPSOCH_2$), 7.35-7.46 (6H, m, aromatic), 7.62-7.66 (4H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.2, 24.9, 26.4, 26.9, 27.4, 37.3, 40.9, 42.5, 50.2, 62.8, 127.7, 129.7, 133.7, 135.5, 210.1, 210.5; HRMS (ESI) calcd for $C_{26}H_{34}O_3SiNa$ 445.2175 ($M+Na^+$), found 445.2169.

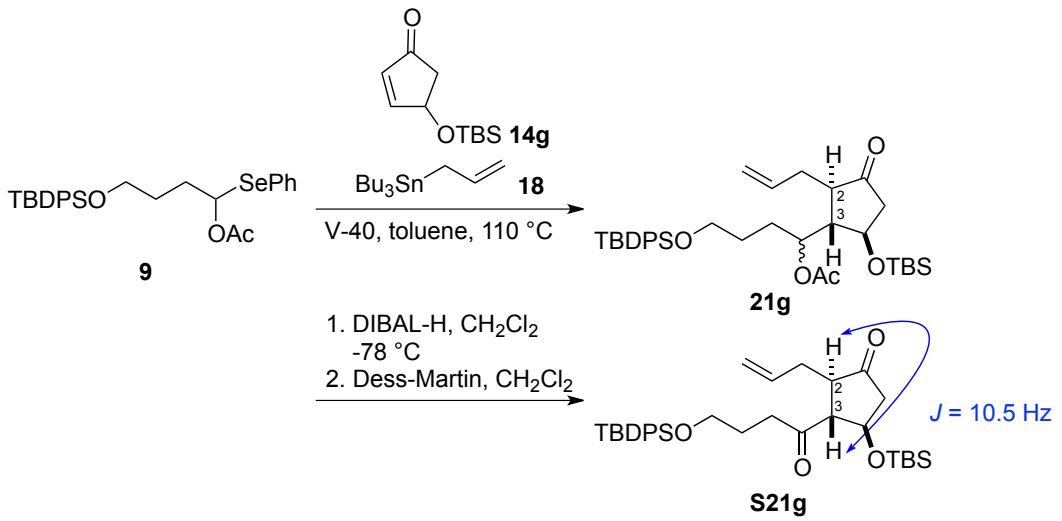
6. Three-component radical reaction in Table 3



Compound 21e. A solution of O,Se-acetal **9** (49 mg, 0.093 mmol), **14e** (39 μL, 0.48 mmol) and AIBN (3.1 mg, 0.019 mmol) in benzene (0.47 mL) was degassed by freeze-thaw procedure (x 3). The mixture was heated to 80 °C and degassed solution of allyltributytin **18** (185 μL, 0.60 mmol) and AIBN (2.8 mg, 0.017 mmol) in benzene (0.47 mL) was added with a syringe pump over 3 h. After additional 1 h at 80 °C, the reaction mixture was cooled to room temperature and concentrated. The residue was purified by flash chromatography [a column consecutively packed with silica gel 15 g and 10% (w/w) KF contained silica gel 5 g, hexane/EtOAc 20:1 to 5:1] to afford **21e** (31 mg, 0.063 mmol) in 68% yield as a 1:1 epimeric mixture at the acetoxy-substituted carbon center: ^{1}H NMR of a 1:1 epimeric mixture of **21e** (400 MHz, $CDCl_3$) δ 1.05 (9H, s, *t*-Bu of TBDPS), 1.50-1.78 (5H, m), 1.87-2.55 (7H, m), 2.039 (3H x 1/2, s, Ac), 2.041 (3H x 1/2, s, Ac), 3.61-3.73 (2H, m, $TBDPSOCH_2$), 4.99-5.11 (3H, m, CH_2=CH, C*H*OAc), 5.58-5.72 (1H, m, CH_2=C*H*), 7.35-7.45 (6H, m, aromatic), 7.63-7.67 (4H, m, aromatic); HRMS (ESI) calcd for $C_{30}H_{40}O_4SiNa$ 515.2594 ($M+Na^+$), found 515.2580.
Since the epimeric mixture of **21e** was inseparable, the structure of the product was established from the NMR data of diketone **S21e**, which was prepared through

deacetylation and subsequent oxidation.

Diketone S21e. According to the general procedure D, diketone **S21e** (7.4 mg, 0.016 mmol) was synthesized in 60% yield over 2 steps from a 1:1 epimeric mixture of **21e** (14 mg, 0.028 mmol) by using K_2CO_3 (12 mg, 0.087 mmol) in MeOH (0.55 mL) for the first step, Dess-Martin periodinane (23 mg, 0.054 mmol) in CH_2Cl_2 (0.55 mL) for the second steps. The crude was purified by flash chromatography (silica gel 2 g, hexane/EtOAc 8:1): colorless oil; IR (neat) ν_{max} 2929, 2856, 1741, 1711, 1111 cm^{-1}; ^1H NMR (400 MHz, C_6D_6) δ 1.12-1.27 (1H, m, $COCH_2CH_AH_B$), 1.17 (9H, s, *t*-Bu of TBDPS), 1.49 (1H, dddd, *J* = 11.9, 9.1, 6.8, 2.3 Hz, $COCH_2CH_AH_B$), 1.60 (1H, ddd, *J* = 18.3, 10.5, 9.1 Hz, $COCH_AH_B$), 1.83 (2H, quintet, *J* = 6.9 Hz, $TBDPSOCH_2CH_2$), 1.94 (1H, br dd, *J* = 18.3, 9.2 Hz, $COCH_AH_B$), 2.11 (1H, dt, *J* = 14.2, 7.4 Hz, $CH_2{=}CHCH_AH_B$), 2.21 (1H, dt, *J* = 18.3, 6.9 Hz, $TBDPSOCH_2CH_2CH_AH_B$), 2.31 (1H, dt, *J* = 18.3, 6.9 Hz, $TBDPSOCH_2CH_2CH_AH_B$), 2.34 (1H, td, *J* = 10.6, 6.8 Hz, $CH_2{=}CHCH_2CHCHCO$), 2.43 (1H, br dt, *J* = 14.2, 6.4 Hz, $CH_2{=}CHCH_AH_B$), 2.54 (1H, m, $CH_2{=}CHCH_2CH$), 3.62 (2H, t, *J* = 6.1 Hz, $TBDPSOCH_2$), 4.87 (1H, d, *J* = 10.0 Hz, $CH_AH_B{=}CH$), 4.90 (1H, dd, *J* = 17.1, 1.4 Hz, $CH_AH_B{=}CH$), 5.59 (1H, dddd, *J* = 17.1, 10.0, 7.2, 7.2 Hz, $CH_2{=}CH$), 7.21-7.25 (6H, m, aromatic), 7.73-7.77 (4H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.2, 25.0, 26.3, 26.8, 33.2, 37.6, 38.9, 50.6, 52.8, 62.8, 117.4, 127.7, 129.7, 133.7, 135.2, 135.5, 210.8, 217.4; HRMS (ESI) calcd for $C_{28}H_{36}O_3SiNa$ 471.2331 ($M+Na^+$), found 471.2325.



Compound 21g. A solution of O,Se-acetal **9** (51 mg, 0.097 mmol), **14g** (105 mg, 0.50 mmol) and AIBN (3.2 mg, 0.020 mmol) in toluene (0.50 mL) was degassed by freeze-thaw procedure (x 3). The mixture was heated to 110 °C and degassed solution of allyltributyltin **18** (190 μL, 0.61 mmol) and AIBN (3.2 mg, 0.020 mmol) in toluene

(0.50 mL) was added with a syringe pump over 3 h. After additional 1 h at 110 °C, the reaction mixture was cooled to room temperature and concentrated. The residue was purified by flash chromatography [a column consecutively packed with silica gel 15 g and 10% (w/w) KF contained silica gel 5 g, 10 g, hexane/EtOAc 20:1] to afford **21g** (28 mg, 0.45 mmol) in 46% yield as a 1:1 epimeric mixture at the acetoxy-substituted carbon center: ^{1}H NMR of a 1:1 epimeric mixture of **21g** (400 MHz, $CDCl_3$) δ 0.04 (3H x 1/2, s, C*H*$_3$ of TBS), 0.05 (3H x 1/2, s, C*H*$_3$ of TBS), 0.07 (3H x 1/2, s, C*H*$_3$ of TBS), 0.08 (3H x 1/2, s, C*H*$_3$ of TBS), 0.871 (9H x 1/2, s, *t*-Bu of TBS), 0.874 (9H x 1/2, s, *t*-Bu of TBS), 1.05 (9H, s, *t*-Bu of TBDPS), 1.50-1.78 (4H, m), 2.01 (3H x 1/2, s, Ac), 2.03 (3H x 1/2, s, Ac), 2.15-2.70 (6H, m), 3.60-3.71 (2H, m, TBDPSOC*H*$_2$), 4.18 (1H x 1/2, ddd, *J* = 6.2, 4.6, 4.6 Hz, TBSOC*H*), 4.32 (1H x 1/2, ddd, *J* = 6.2, 4.6, 4.6 Hz, TBSOC*H*), 4.95-5.15 (3H, m, C*H*$_2$=CH, C*H*OAc), 5.64-5.76 (1H, m, CH$_2$=C*H*), 7.35-7.45 (6H, m, aromatic), 7.64 (4H, dd, *J* = 7.6, 1.1 Hz, aromatic); HRMS (ESI) calcd for $C_{36}H_{54}O_5Si_2Na$ 645.3407 (M+Na$^+$), found 645.3388.

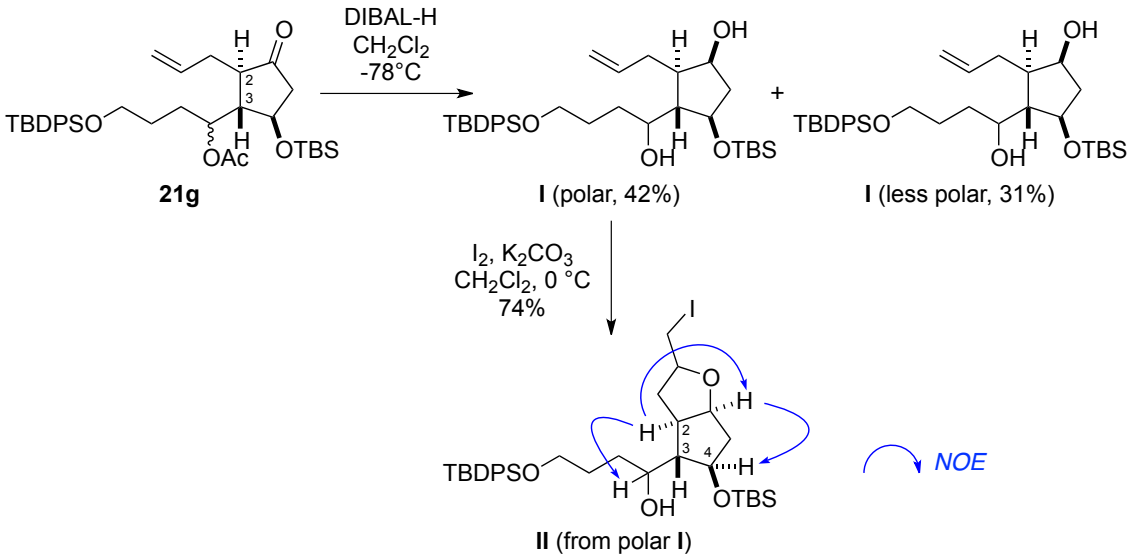
Since the epimeric mixture of **21g** was inseparable, the structure of the product was established from the NMR data of diketone **S21g**, which was prepared through deacetylation and subsequent oxidation.

Diketone S21g. DIBAL-H (1.5 M in toluene, 26 μL, 0.039 mmol) was added to a solution of **21g** (4.9 mg, 7.9 μmol) in CH_2Cl_2 (0.79 mL) at -78 °C. The reaction mixture was stirred at -78 °C for 10 min and at 0 °C for 10 min. Saturated aqueous Rochelle's salt (1 mL) was added, and then the resultant mixture was stirred at room temperature for 1 h. The solution was extracted with CH_2Cl_2 (3 mL x 3) and the combined organic layers were dried over Na_2SO_4, filtered, and concentrated to give the crude diol, which was used to the next reaction without further purification.

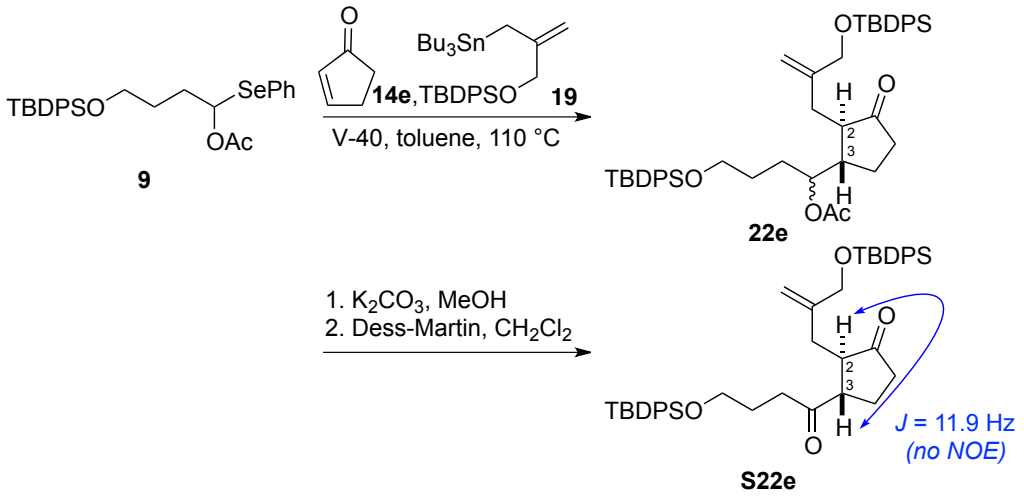
Dess-Martin periodinane (17 mg, 0.039 mmol) was added to a solution of the crude diol in CH_2Cl_2 (0.79 mL) at 0 °C. The reaction mixture was stirred at room temperature for 1 h, and saturated aqueous $Na_2S_2O_3$ (1 mL) was added. The resultant mixture was extracted with CH_2Cl_2 (3 mL x 3) and the combined organic layers were dried over Na_2SO_4, filtered, and concentrated. The residue was purified by flash chromatography (silica gel 2 g, hexane/EtOAc 20:1) to afford diketone **S21g** (2.5 mg, 4.3 μmol) in 55% yield over 2 steps: colorless oil; IR (neat) ν_{max} 2955, 2929, 2857, 1747, 1712 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ -0.01 (3H, s, C*H*$_3$ of TBS), 0.02 (3H, s, C*H*$_3$ of TBS), 0.85 (9H, s, *t*-Bu of TBS), 1.04 (9H, s, *t*-Bu of TBDPS), 1.75-1.91 (2H, m, TBDPSOCH$_2$C*H*$_2$), 2.16-2.21 (1H, m, COC*H*$_A$H$_B$COTBS), 2.22 (1H, dd, *J* = 13.8, 8.2 Hz, CH$_2$=CHC*H*$_A$H$_B$), 2.48 (1H, m, CH$_2$=CHCH$_A$*H*$_B$), 2.61-2.74 (4H, m, COCH$_A$*H*$_B$COTBS, COC*H*CH$_2$, TBDPSOCH$_2$CH$_2$C*H*$_2$), 3.05 (1H, dd, *J* = 10.5, 7.9 Hz,

TBSOCHC*H*), 3.66 (2H, t, *J* = 7.2 Hz, TBDPSOCH_2), 4.32 (1H, q, *J* = 7.9 Hz, TBSOC*H*), 4.98 (1H, d, *J* = 10.0 Hz, C$H_A$$H_B$=CH), 5.02 (1H, dd, *J* = 16.9, 0.9 Hz, CH$_A$$H_B$=CH), 5.59 (1H, dddd, *J* = 16.9, 10.0, 8.2, 6.6 Hz, CH$_2$=C*H*), 7.35-7.46 (6H, m, aromatic), 7.63-7.67 (4H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ -5.1, -4.7, 17.8, 19.2, 25.6, 26.1, 26.8, 33.3, 42.2, 47.7, 51.7, 61.4, 63.0, 72.1, 117.6, 127.6, 129.6, 133.7, 135.2, 135.5, 211.2, 213.6; HRMS (ESI) calcd for $C_{34}H_{50}O_4Si_2Na$ 601.3145 (M+Na$^+$), found 601.3136.

The C2, 3, 4-stereochemistries of 2,3-*trans*-3,4-*trans*-trisubstituted cyclopentanone of **21g** was determined from the NOE experiments after transformation of **21g** to compound **II** through DIBAL-H reduction and iodo-etherification.



Compound II: colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.06 (9H, s, *t*-Bu of TBDPS), 1.49-1.91 (7H, m, C*H*CHOH, COCHC$H_A$$H_B$, COCHCHC$H_A$$H_B$, TBDPSOCH$_2CH_2CH_2$), 1.97 (1H, ddd, *J* = 12.2, 5.4, 1.6 Hz, COCHCHCH$_A$$H_B$), 2.28 (1H, dddd, *J* = 7.9, 6.8, 6.8, 1.6 Hz, COCHC*H*), 2.40 (1H, ddd, *J* = 12.6, 6.8, 6.8 Hz, COCHCH$_A$$H_B$), 3.23 (1H, dd, *J* = 9.9, 6.1 Hz, C$H_A$$H_B$I), 3.28 (1H, dd, *J* = 9.9, 5.0 Hz, CH$_A$$H_B$I), 3.44 (1H, br, O*H*), 3.63 (1H, dd, *J* = 9.1, 9.1 Hz, C*H*OH), 3.72 (2H, m, TBDPSOCH_2), 3.96 (1H, ddd, *J* = 8.9, 8.0, 6.8 Hz, C*H*OTBS), 4.09 (1H, m, C*H*CH$_2$I), 4.59 (1H, ddd, *J* = 6.8, 6.8, 6.8 Hz, COC*H*CH), 7.37-7.48 (6H, m, aromtic), 7.64-7.69 (4H, m, aromatic); HRMS (ESI) calcd for $C_{28}H_{39}INaO_4Si$ 617.1555 (M+Na$^+$), found 617.1555.

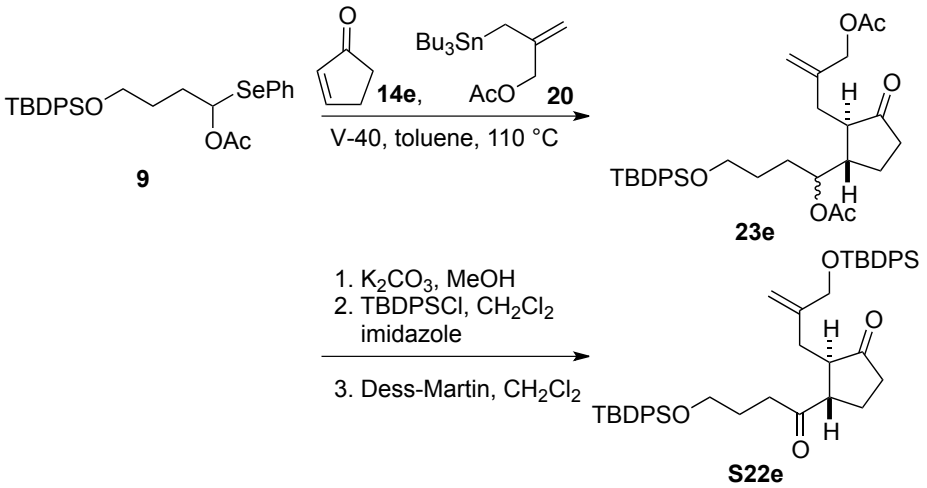


Compound 22e. A solution of O,Se-acetal **9** (52 mg, 0.099 mmol), **14e** (42 μL, 0.50 mmol) and V-40 (5.1 mg, 0.021 mmol) in toluene (0.50 mL) was degassed by freeze-thaw procedure (x 3). The mixture was heated to 110 °C and a degassed solution of allyltributytin **19**[8] (365 mg, 0.60 mmol) and V-40 (5.1 mg, 0.021 mmol) in toluene (0.50 mL) was added with a syringe pump over 3 h. After additional 2 h at 110 °C, the reaction mixture was cooled to room temperature and concentrated. The residue was purified by flash chromatography [a column consecutively packed with silica gel 15 g and 10% (w/w) KF contained silica gel 5 g, hexane/EtOAc 20:1 to 5:1] to afford **22e** (51 mg, 0.067 mmol) in 68% yield as a 1:1 epimeric mixture at the acetoxy-substituted carbon center: ^{1}H NMR of a 1:1 epimeric mixture of **22e** (400 MHz, $CDCl_3$) δ 1.041 (9H x 1/2, s, *t*-Bu of TBDPS), 1.049 (9H x 1/2, s, *t*-Bu of TBDPS), 1.06 (9H, s, *t*-Bu of TBDPS), 1.42-2.37 (12H, m), 1.92 (3H x 1/2, s, Ac), 1.96 (3H x 1/2, s, Ac), 3.59-3.70 (2H, m, TBDPSOCH_2), 3.84 (1H x 1/2, d, J = 15.1 Hz, TBDOPSOCH_AH$_B$C=CH$_2$), 3.92 (1H x 1/2, d, J = 15.1 Hz, TBDOPSOCH_AH$_B$C=CH$_2$), 4.04 (1H x 1/2, d, J = 15.1 Hz, TBDOPSOCH$_A$$H_B$C=CH$_2$), 4.08 (1H x 1/2, d, J = 15.1 Hz, TBDOPSOCH_AH$_B$C=CH$_2$), 4.88 (1H x 1/2, s, CH_AH$_B$=C), 4.92 (1H x 1/2, s, CH_AH$_B$=C), 4.93 (1H x 1/2, m, C*H*OAc), 4.99 (1H x 1/2, m, C*H*OAc), 5.31 (1H x 1/2, s, CH$_A$$H_B$=C), 5.34 (1H x 1/2, s, CH$_A$$H_B$=C), 7.32-7.45 (12H, m, aromatic), 7.63-7.68 (8H, m, aromatic); HRMS (ESI) calcd for $C_{47}H_{60}O_5Si_2Na$ 783.3877 (M+Na$^+$), found 783.3893.

Since the epimeric mixture of **22e** was inseparable, the structure of the product was established from the NMR data of diketone **S22e**, which was prepared through deacetylation and subsequent oxidation.

Diketone S22e. According to the general procedure D, diketone **S22e** (18 mg, 0.024 mmol) was synthesized in 60% yield from a 1:1 epimeric mixture of **22e** (32 mg, 0.042

mmol) by using K_2CO_3 (18 mg, 0.13 mmol) in MeOH (0.84 mL) for the first step, Dess-Martin periodinane (36 mg, 0.085 mmol) in CH_2Cl_2 (0.84 mL) for the second steps. The crude was purified by flash chromatography (silica gel 2 g, hexane/EtOAc 10:1): colorless oil; IR (neat) ν_{max} 2931, 2857, 1742, 1713, 1428, 1112 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.049 (9H, s, *t*-Bu of TBDPS), 1.054 (9H, s, *t*-Bu of TBDPS), 1.63 (1H, m, $COCH_2CH_AH_B$), 1.73-1.87 (2H, m, $TBDPSOCH_2CH_2$), 2.01 (1H, dd, J = 18.3, 8.7 Hz, $COCH_AH_B$), 2.06 (1H, dd, J = 8.7, 5.5 Hz, $CH_2{=}CCH_AH_B$), 2.13 (1H, dddd, J = 11.9, 8.7, 6.8, 3.2 Hz, $COCH_2CH_AH_B$), 2.36 (1H, br dd, J = 18.3, 8.7 Hz, $COCH_AH_B$), 2.45-2.55 (2H, m, $CH_2{=}CCH_AH_B$, $TBDPSOCH_2CH_2CH_AH_B$), 2.60-2.70 (2H, m, $CH_2{=}CCH_2CH$, $TBDPSOCH_2CH_2CH_AH_B$), 2.82 (1H, td, J = 11.9, 6.8 Hz, COCHC*H*), 3.65 (2H, t, J = 6.4 Hz, $TBDPSOCH_2$), 3.90 (1H, d, J = 14.6 Hz, $CH_2{=}CCH_AH_BOTBDPS$), 4.03 (1H, d, J = 14.6 Hz, $CH_2{=}CCH_AH_BOTBDPS$), 4.79 (1H, m, $CH_AH_B{=}C$), 5.16 (1H, m, J = 2.0 Hz, $CH_AH_B{=}C$), 7.34-7.45 (12H, m, aromatic), 7.62-7.68 (8H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.19, 19.24, 25.0, 26.3, 26.81, 26.84, 32.4, 37.2, 38.8, 49.6, 53.1, 62.8, 65.5, 111.5, 127.6, 129.6, 133.4, 133.5, 133.70, 133.72, 135.49, 135.53, 145.6, 210.6, 217.4; HRMS (ESI) calcd for $C_{45}H_{56}O_4Si_2Na$ 739.3615 ($M+Na^+$), found 739.3604.



Compound 23e. A solution of O,Se-acetal **9** (206 mg, 0.39 mmol), **14e** (160 μL, 2.0 mmol) and V-40 (19 mg, 0.08 mmol) in toluene (2.0 mL) was degassed by freeze-thaw procedure (x 3). The mixture was heated to 110 °C and a degassed solution of allyltributyltin **20**[9] (945 mg, 2.4 mmol) and V-40 (19 mg, 0.08 mmol) in toluene (1.9 mL) was added with a syringe pump over 3 h. After additional 2 h at 110 °C, the reaction mixture was cooled to room temperature and concentrated. The residue was purified by flash chromatography [a column consecutively packed with silica gel 15 g

and 10% (w/w) KF contained silica gel 5 g, hexane/EtOAc 10:1 to 3:1] to afford **23e** (129 mg, 0.23 mmol) in 59% yield as a 1:1 epimeric mixture at the acetoxy-substituted carbon center: ^{1}H NMR of a 1:1 epimeric mixture of **23e** (400 MHz, $CDCl_3$) δ 1.05 (9H, s, *t*-Bu of TBDPS), 1.45-1.86 (6H, m), 1.97-2.45 (6H, m), 2.03 (3H, s, Ac), 2.070 (3H x 1/2, s, Ac), 2.075 (3H x 1/2, s, Ac), 3.60-3.72 (2H, m, TBDPSOCH_2), 4.44 (2H x 1/2, s, CH_2OAc), 4.48 (2H x 1/2, s, CH_2OAc), 4.94 (1H x 1/2, s, CH_AH$_B$=C), 4.98 (1H x 1/2, s, CH_AH$_B$=C), 5.08 (1H x 1/2, s, CH$_A$$H_B$=C), 5.12 (1H x 1/2, s, CH$_A$$H_B$=C), 4.95-5.10 (1H, m, C*H*OAc), 7.35-7.46 (6H, m, aromatic), 7.62-7.67 (4H, m, aromatic); HRMS (ESI) calcd for $C_{33}H_{44}O_6SiNa$ 587.2805 (M+Na$^+$), found 587.2814.

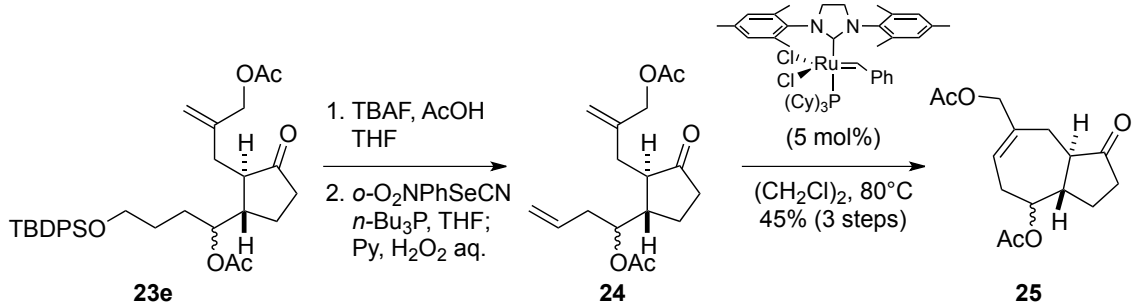
Since the epimeric mixture of **23e** was inseparable, the structure of the product was established from the NMR data of diketone **S22e**, which was prepared through deacetylation and subsequent oxidation.

Diketone S22e. A 1:1 epimeric mixture of **23e** (43 mg, 0.076 mmol) and K_2CO_3 (32 mg, 0.23 mmol) in MeOH (1.5 mL) was stirred for 15 h at room temperature. Saturated aqueous NH_4Cl (1 mL) was added, and the mixture was extracted with CH_2Cl_2 (5 mL x 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated to give the crude alcohol, which was used to the next reaction without further purification.

A mixture of the crude alcohol, TBDPSCl (23 μL, 0.090 mmol) and imidazole (13 mg, 0.19 mmol) in CH_2Cl_2 (1.5 mL) was stirred at room temperature for 15 min. Saturated aqueous $NaHCO_3$ (1 mL) was added to the reaction mixture, which was extracted with CH_2Cl_2 (3 mL x 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated to give the crude TBDPS ether, which was used to the next reaction without further purification.

Dess-Martin periodinane (65 mg, 0.15 mmol) was added to a solution of the crude alcohol in CH_2Cl_2 (1.5 mL) at 0 °C. After 1 h at room temperature, saturated aqueous $Na_2S_2O_3$ (1 mL) was added. The mixture was extracted with CH_2Cl_2 (2 mL x 3) and the combined organic layers were dried over Na_2SO_4, filtered, and concentrated. The residue was purified by flash chromatography (silica gel 4 g, hexane/EtOAc 8:1) to afford diketone **S22e** (39 mg, 0.054 mmol) in 71% yield. Analytical data of synthesized ketone was identical with those of **S22e** obtained from **22e**.

7. Construction of bicyclo[5.3.0]decene structure of zedoarondiol



Compound 25. A mixture of **23e** (185 mg, 0.33 mmol), TBAF (1.0 M in THF, 650 μL, 0.65 mmol) and AcOH (75 μL, 1.32 mmol) in THF (5 mL) was stirred at room temperature for 36 h. Saturated aqueous NH_4Cl (30 mL) was added, and the mixture was extracted with EtOAc (20 mL x 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The crude was roughly purified by flash chromatography (silica gel 5 g, hexane/EtOAc 1:1 to EtOAc/2-PrOH 7:1) to afford the crude alcohol, which was used for the next reaction without further purification.

n-Bu_3P (100 μL, 0.41 mmol) was added to a mixture of the crude alcohol and *o*-$NO_2PhSeCN$ (73 mg, 0.32 mmol) in THF (0.5 mL). The reaction mixture was stirred at room temperature for 1 h, and then *n*-Bu_3P (100 μL, 0.41 mmol) and *o*-$NO_2PhSeCN$ (72 mg, 0.32 mmol) were added. The reaction mixture was stirred for further 1 h, and cooled to 0 °C. Pyridine (161 μL, 1.98 mmol) and H_2O_2 (100 μL, 8.8 M in H_2O, 0.88 mmol) were successively added, and the reaction mixture was stirred at room temperature for 13 h. Saturated aqueous $NaHCO_3$ (5 mL) was added and the resultant mixture was extracted with Et_2O (10 mL x 3). The combined organic layers were dried over Na_2SO_4, filtered and concentrated. The crude was purified by flash chromatography (silica gel 5 g, CH_2Cl_2/EtOAc 20:1 to hexane/EtOAc 3:1) to afford diene **24**.

A solution of diene **24** in $(CH_2Cl)_2$ (7 mL) was degassed by freeze-thaw (x 3). The solution was heated to 80 °C and degassed solution of Grubbs' 2nd generation catalyst (14 mg, 0.016 mmol) in $(CH_2Cl)_2$ (4 mL) was added with a syringe pump over 3 h. After the addition of the catalyst, the reaction mixture was further stirred for 30 min at 80 °C, cooled to room temperature and concentrated. The residue was purified by flash chromatography (silica gel 5 g, hexane/EtOAc 4:1 to 2:1) to afford **25** (42 mg, 0.15 mmol) in 45% yield over 3 steps. Analytically pure **25 polar** and **25 less polar** were obtained by separation from the epimeric mixture of **25** using flash chromatography (silica gel 5 g, hexane/EtOAc 4:1). **25 polar**: colorless oil; IR (neat) ν_{max} 2941, 1735, 1375, 1236 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.59-1.70 (1H, m,

$COCH_2CH_AH_B$), 1.90-2.20 (5H, m, $COCHCH_AH_B$, $COCHCH$, $COCH_2CH_AH_B$, $COCH_AH_B$), 2.06 (3H, s, Ac), 2.07 (3H, s, Ac), 2.26 (1H, dq, J = 16.0, 1.9 Hz, $C{=}CHCH_AH_B$), 2.39 (1H, dd, J = 19.7, 9.2 Hz, $COCH_AH_B$), 2.69 (1H, d, J = 15.6 Hz, $COCHCH_AH_B$), 2.78 (1H, ddd, J = 16.0, 8.7, 6.0 Hz, $C{=}CHCH_AH_B$), 4.47 (1H, d, J = 14.2 Hz, $AcOCH_AH_B$), 4.51 (1H, d, J = 14.2 Hz, $AcOCH_AH_B$), 5.20 (1H, d, J = 6.0 Hz, AcOC*H*), 5.59 (1H, m, C=C*H*); ^{1}H NMR (400 MHz, C_6D_6) δ 0.98-1.25 (2H, m, COCHC*H*, $COCH_2CH_AH_B$), 1.26-1.34 (1H, m, $COCH_2CH_AH_B$), 1.55-1.70 (2H, m, $COCH_AH_B$, $COCHCH_AH_B$), 1.64 (3H, s, Ac), 1.65 (3H, s, Ac), 1.75 (1H, dq, J = 16.0, 1.9 Hz, $C{=}CHCH_AH_B$), 1.84 (1H, dd, J = 11.9 Hz, $COCHCH_2$), 1.92 (1H, dd, J = 18.3, 7.3 Hz, $COCH_AH_B$), 2.57 (1H, ddd, J = 16.0, 8.7, 6.0 Hz, $C{=}CHCH_AH_B$), 2.81 (1H, d, J = 15.6 Hz, $COCHCH_AH_B$), 4.39 (1H, d, J = 14.2 Hz, $AcOCH_AH_B$), 4.43 (1H, d, J = 14.2 Hz, $AcOCH_AH_B$), 4.92 (1H, 1H, d, J = 6.0 Hz, C*H*OAc), 5.38 (1H, m, C=C*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.9, 21.1, 23.4, 28.8, 31.1, 36.0, 46.8, 50.5, 69.5, 69.6, 124.2, 137.3, 170.7, 170.8, 217.1; HRMS (ESI) calcd for $C_{15}H_{20}O_5Na$ 303.1208 ($M+Na^+$), found 303.1209. **25 less polar**: colorless oil; IR (neat) ν_{max} 2937, 1737, 1370, 1237 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.46-1.60 (1H, m, $COCH_2CH_AH_B$), 1.75 (1H, dd, J = 12.8, 11.0 Hz, $COCHCH_2$), 1.94 (1H, dd, J = 15.8, 12.8 Hz, $CH_AH_BC{=}CH$), 1.98-2.34 (4H, m, $COCH_AH_BCH_AH_BCH$, $C{=}CHCH_AH_B$), 2.07 (3H, s, Ac), 2.09 (3H, s, Ac), 2.39 (1H, dd, J = 19.2, 8.7 Hz, $COCH_AH_B$), 2.48 (1H, ddd, J = 14.2, 9.1, 2.7 Hz, $C{=}CHCH_AH_B$), 2.62 (1H, d, J = 15.8 Hz, $CH_AH_BC{=}CH$), 4.45 (1H, d, J = 14.2 Hz, $AcOCH_AH_B$), 4.49 (1H, d, J = 14.2 Hz, $AcOCH_AH_B$), 4.68 (1H, ddd, J = 10.5, 10.5, 2.7 Hz, C*H*OAc), 5.78 (1H, m, C=C*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.9, 21.1, 25.0, 27.7, 33.5, 35.6, 51.9, 52.0, 69.2, 75.0, 124.2, 138.7, 170.3, 170.8, 216.0; HRMS (ESI) calcd for $C_{15}H_{20}O_5Na$ 303.1208 ($M+Na^+$), found 303.1204.

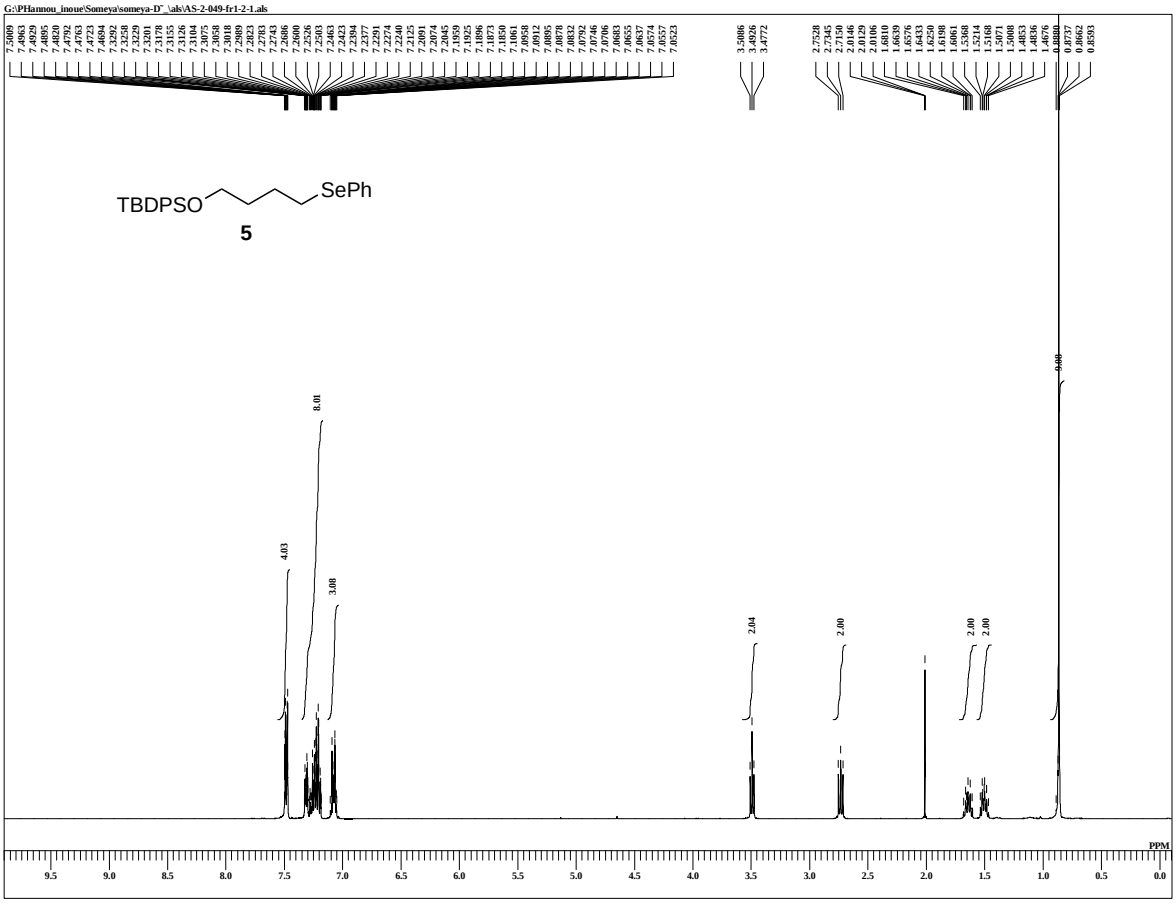
8. References

1. Freeman, F.; Kim, D. S. H. L., Rodoriguez, E. *J. Org. Chem.* **1992**, *57*, 1722-1727.
2. Trost, B. M.; Moeller, K. D. *Heterocycles* **1989**, *28*, 321-331.
3 Fadnavis, N. W.; Reddipalli, G. S.; Ramakrishna, G.; Mishra, M. K.; Sheelu, G. *Synthesis* **2009**, 557-560.
4. Higuchi, H.; Otsubo, T.; Ogura, F.; Yamaguchi, H.; Sakata, Y.; Misumi, S. *Bull. Chem. Soc. Jpn.,* **1982**, *55*, 182-187.
5. Middleton, D. S.; Simpkins, N. S. Terrett, N. K. *Tetrahedron Lett.* **1989**, *30*, 3865-3868.
6. Sharpless, K. B.; Young, M. W. *J. Org. Chem.* **1975**, *40*, 947-949.
7 Depezay, J.-C.; Merrer, Y. L.; *Carbohydr. Res.* **1980**, *83*, 51-62.
8. a) Weigand, S.; Bruckner, R. *Synthesis* **1996**, 475-482. b) Vloon, W. J.; van den

Bos, J. C.; Koomen, G.-J.; Pandit, U. K. *Tetrahedron* **1992**, *48*, 8317-8328.
9. Trost, B. M.; Bonk, P. J. *J. Am. Chem. Soc.* **1985**, *107*, 1778-1781.

AS-2-049-fr1-2

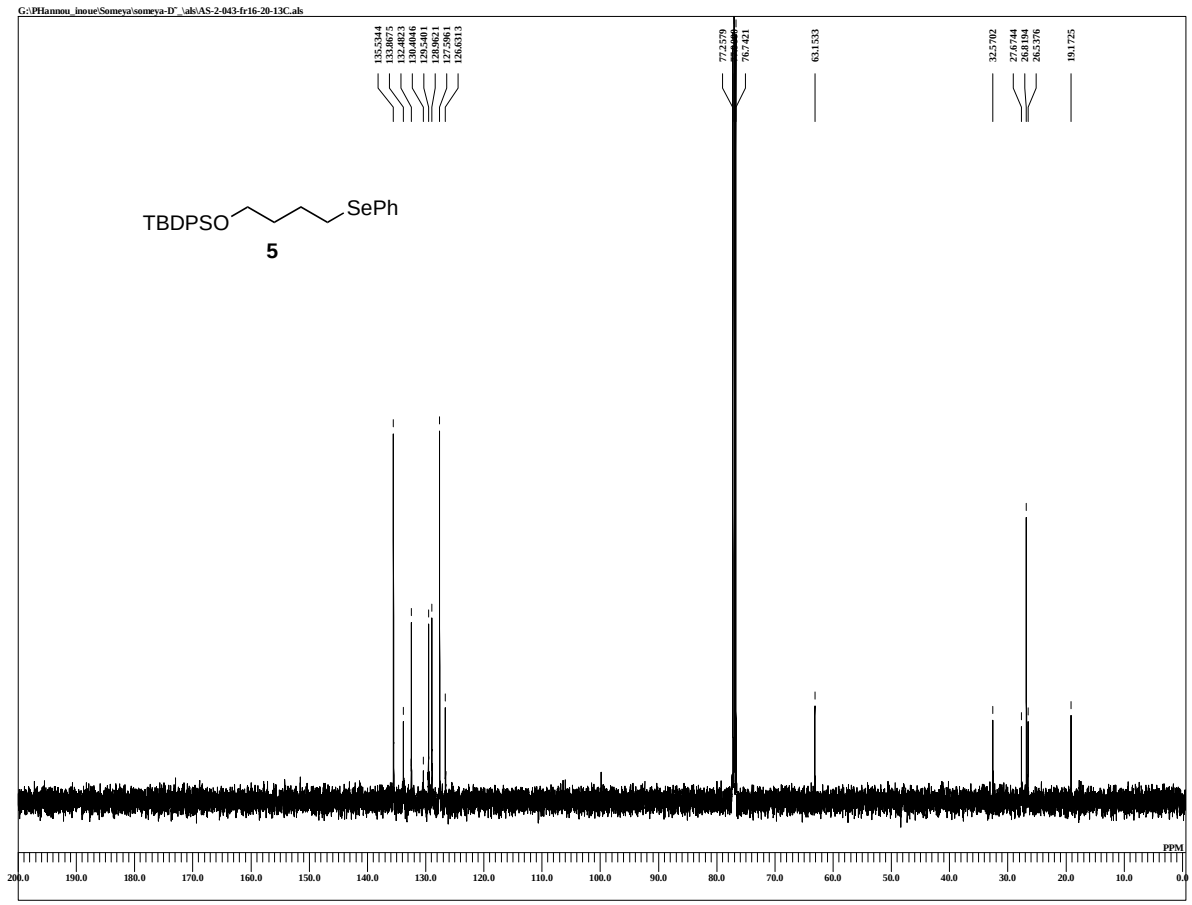
G:\PHannou_inoue\Someya\someya-D^_\als\AS-2-049-fr1-2-1.als



DFILE AS-2-049-fr1-2-1.als
COMNT AS-2-049-fr1-2
DATIM 06-02-2009 22:59:48
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.50 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 26214
SPO 26214
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 34
BF 0.01 Hz
T1 0.00
T2 0.00
T3 70.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE AS-2-049-fr1-2-1.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 17.3 c
SLVNT CDCL3
EXREF 7.26 ppm

AS-2-043-fr16-20-13C

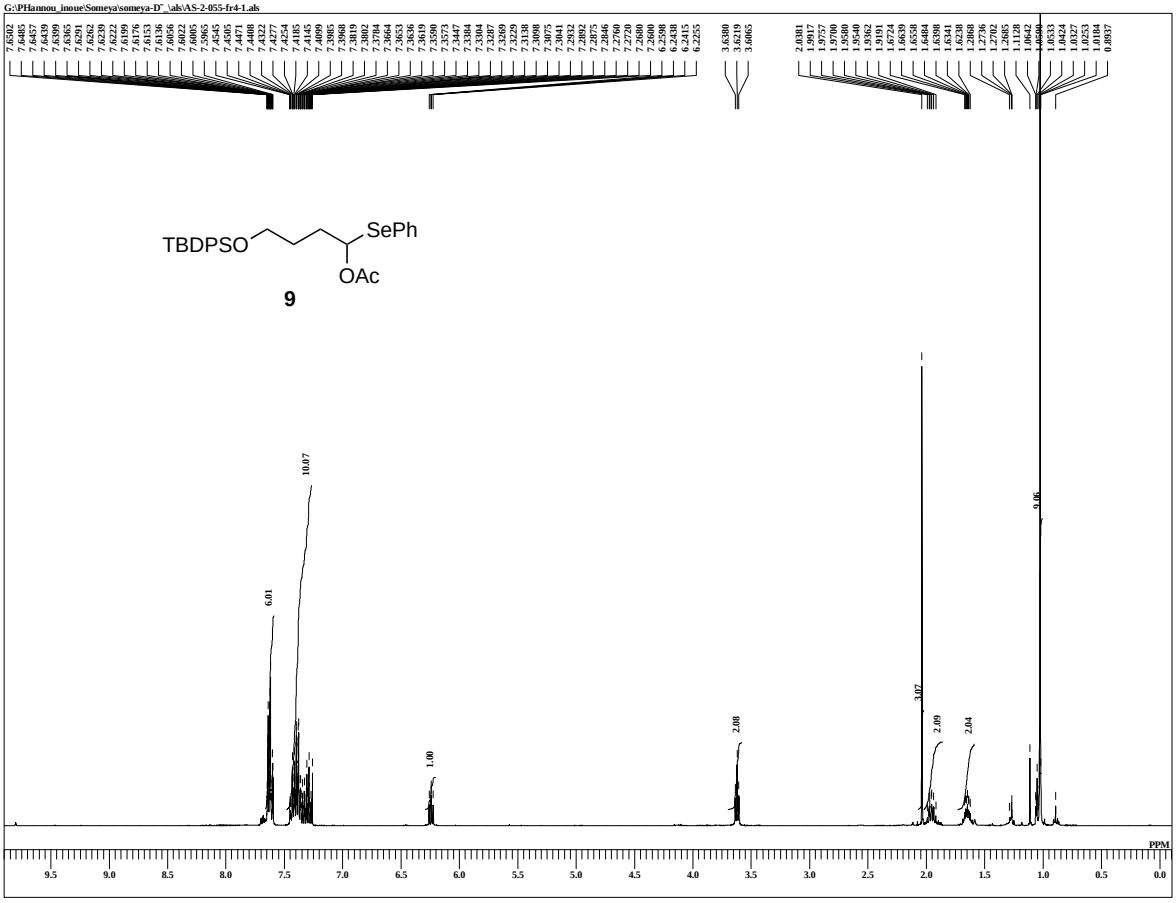
G:\PHannou_inoue\Someya\someya-D^_\als\AS-2-043-fr16-20-13C.als



DFILE AS-2-043-fr16-20-13C.als
COMNT AS-2-043-fr16-20-13C
DATIM 06-02-2009 16:08:04
MENUF
OBNUC 13C
OFR 123.26 MHz
OBFRQ 123.26 MHz
OBSET 2.31 KHz
OBFIN 6.71 Hz
PW1 3.20 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 52428
SPO 52428
TIMES 121
DUMMY 4
FREQU 30863.73 Hz
FLT 155000 Hz
DELAY 21.06 usec
ACQTM 0.8493 sec
PD 2.0000 sec
SCANS 121
ADBIT 16
RGAIN 50
BF 1.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 490.15 MHz
IRSET 9.16 KHz
IRFIN 7.60 Hz
IRRPW 92 usec
IRATN 79
DFILE AS-2-043-fr16-20-13C.als
SF
LKSET 70.30 KHz
LKFIN 32.5 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 19.2 c
SLVNT CDCL3
EXREF 77.00 ppm

AS-2-055-fr4

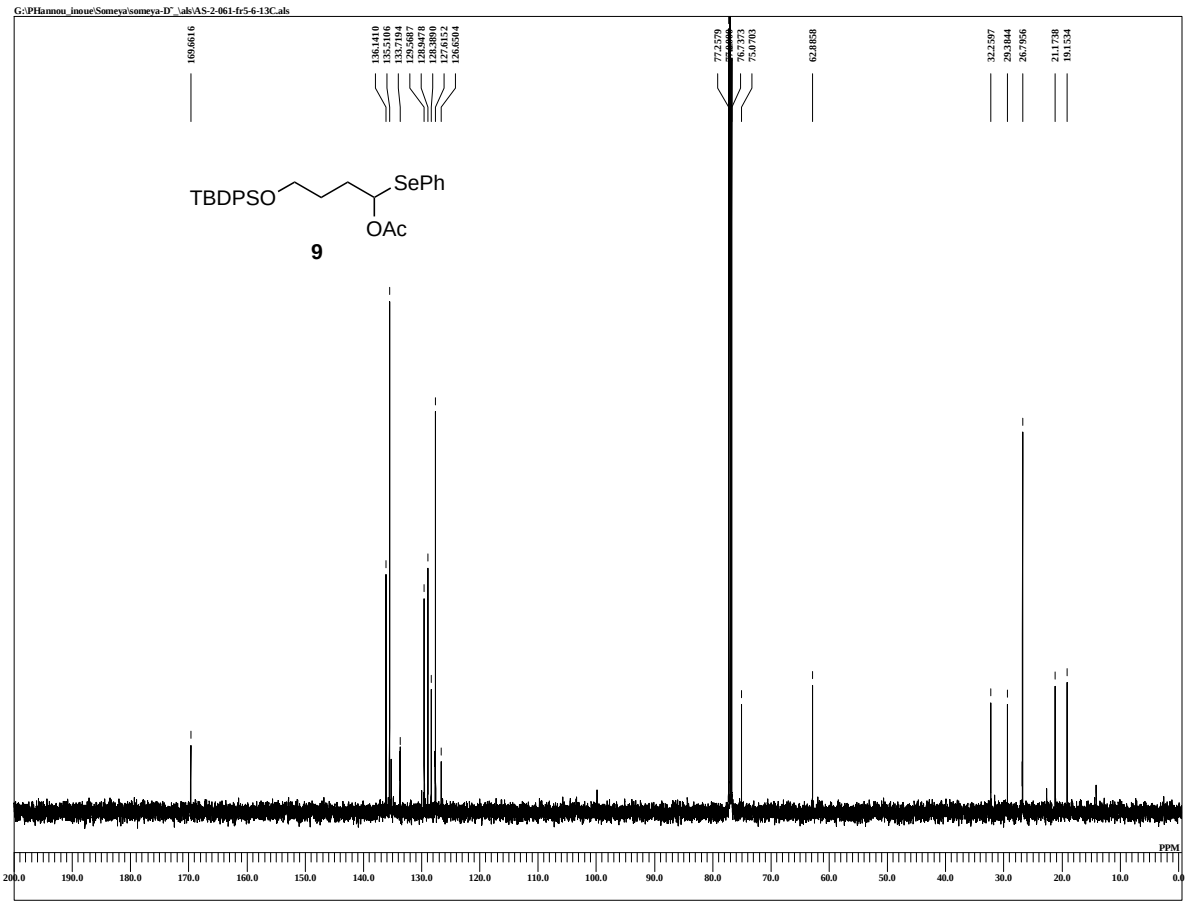
G:\PHannou_inoue\Someya\someya-D^_\als\AS-2-055-fr4-1.als



DFILE AS-2-055-fr4-1.als
COMNT AS-2-055-fr4
DATIM 12-02-2009 23:12:23
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.50 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 26214
SPO 26214
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 30
BF 0.01 Hz
T1 0.00
T2 0.00
T3 70.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE AS-2-055-fr4-1.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 17.5 c
SLVNT CDCL3
EXREF 7.26 ppm

AS-2-061-fr5-6-13C

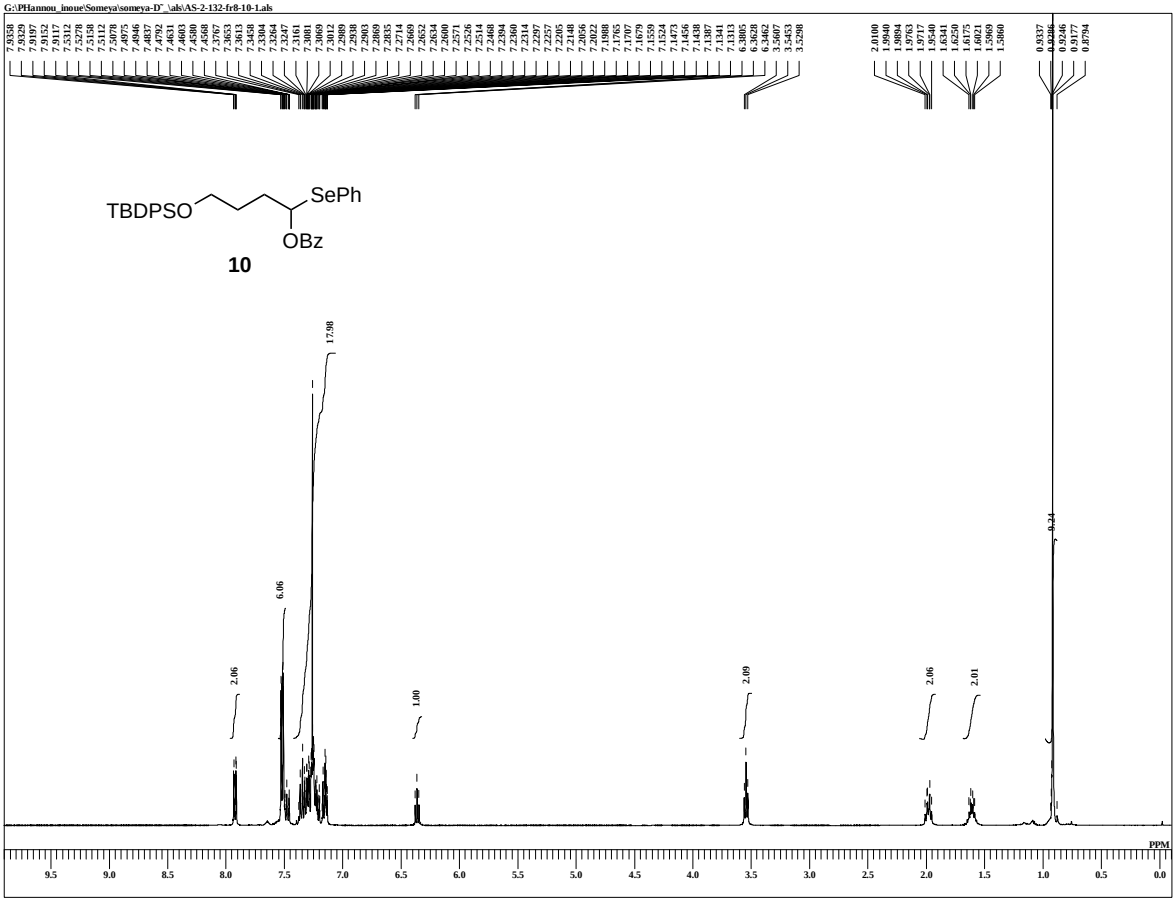
G:\PHannou_inoue\Someya\someya-D^_\als\AS-2-061-fr5-6-13C.als



DFILE AS-2-061-fr5-6-13C.als
COMNT AS-2-061-fr5-6-13C
DATIM 24-04-2009 10:32:08
MENUF
OBNUC 13C
OFR 123.26 MHz
OBFRQ 123.26 MHz
OBSET 2.31 KHz
OBFIN 6.71 Hz
PW1 3.20 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 52428
SPO 52428
TIMES 121
DUMMY 4
FREQU 30863.73 Hz
FLT 155000 Hz
DELAY 21.06 usec
ACQTM 0.8493 sec
PD 2.0000 sec
SCANS 121
ADBIT 16
RGAIN 50
BF 1.00 Hz
T1 0.00
T2 0.00
T3 70.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 490.15 MHz
IRSET 9.16 KHz
IRFIN 7.60 Hz
IRRPW 92 usec
IRATN 79
DFILE AS-2-061-fr5-6-13C.als
SF
LKSET 70.30 KHz
LKFIN 32.5 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.8 c
SLVNT CDCL3
EXREF 77.00 ppm

AS-2-132-fr8-10

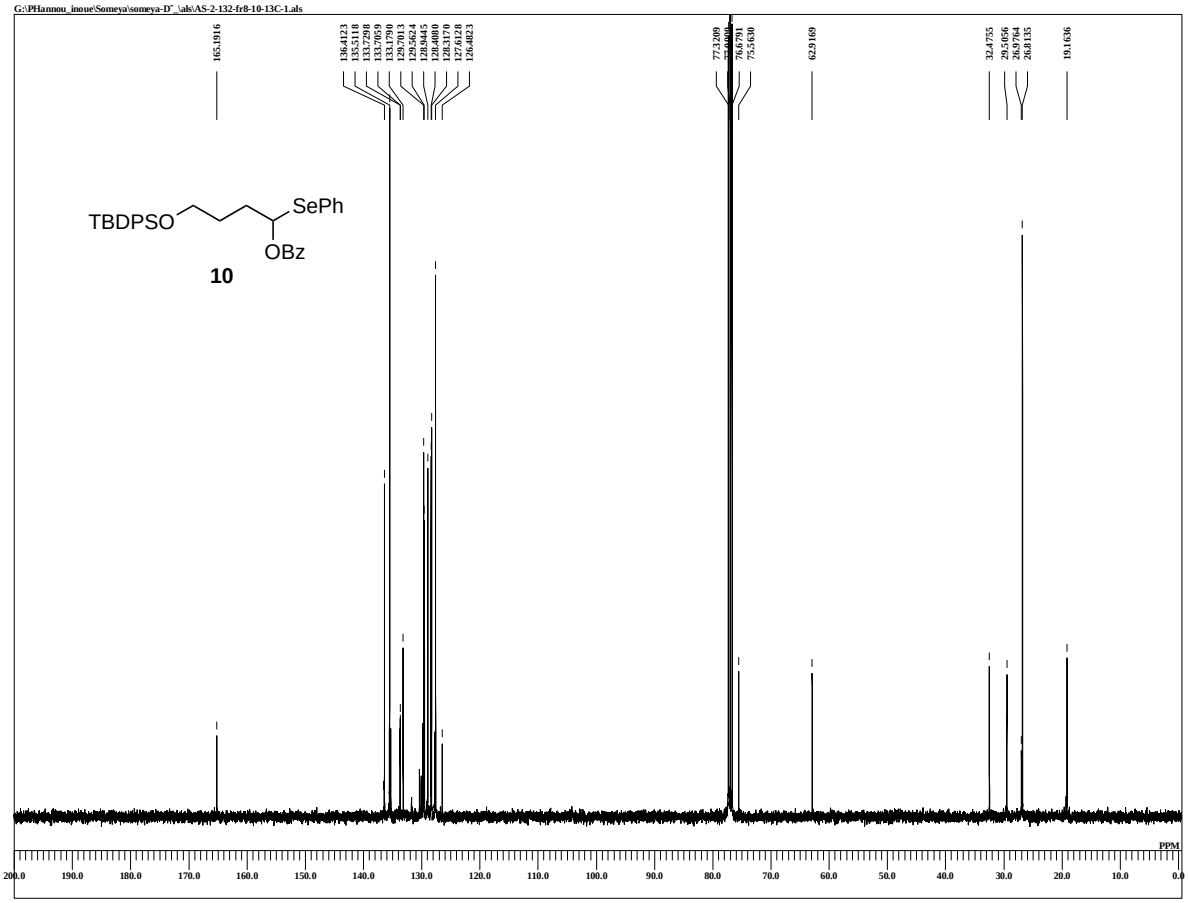
G:\PHannou_inoue\Someya\someya-D^_\als\AS-2-132-fr8-10-1.als




```
DFILE    AS-2-132-fr8-10-1.als
COMNT    AS-2-132-fr8-10
DATIM    26-02-2011 18:08:43
MENUF
OBNUC    1H
OFR      395.88 MHz
OBFRQ    395.88 MHz
OBSET    6.28 KHz
OBFIN    0.87 Hz
PW1      6.50 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    26214
SPO      26214
TIMES    8
DUMMY    1
FREQU    5938.15 Hz
FLT      30000 Hz
DELAY    16.68 usec
ACQTM    2.2073 sec
PD       2.0000 sec
SCANS    8
ADBIT    16
RGAIN    30
BF       0.01 Hz
T1       0.00
T2       0.00
T3       70.00
T4       100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    AS-2-132-fr8-10-1.als
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    20.4 c
SLVNT    CDCL3
EXREF    7.26 ppm
```

AS-2-132-fr8-10-13C

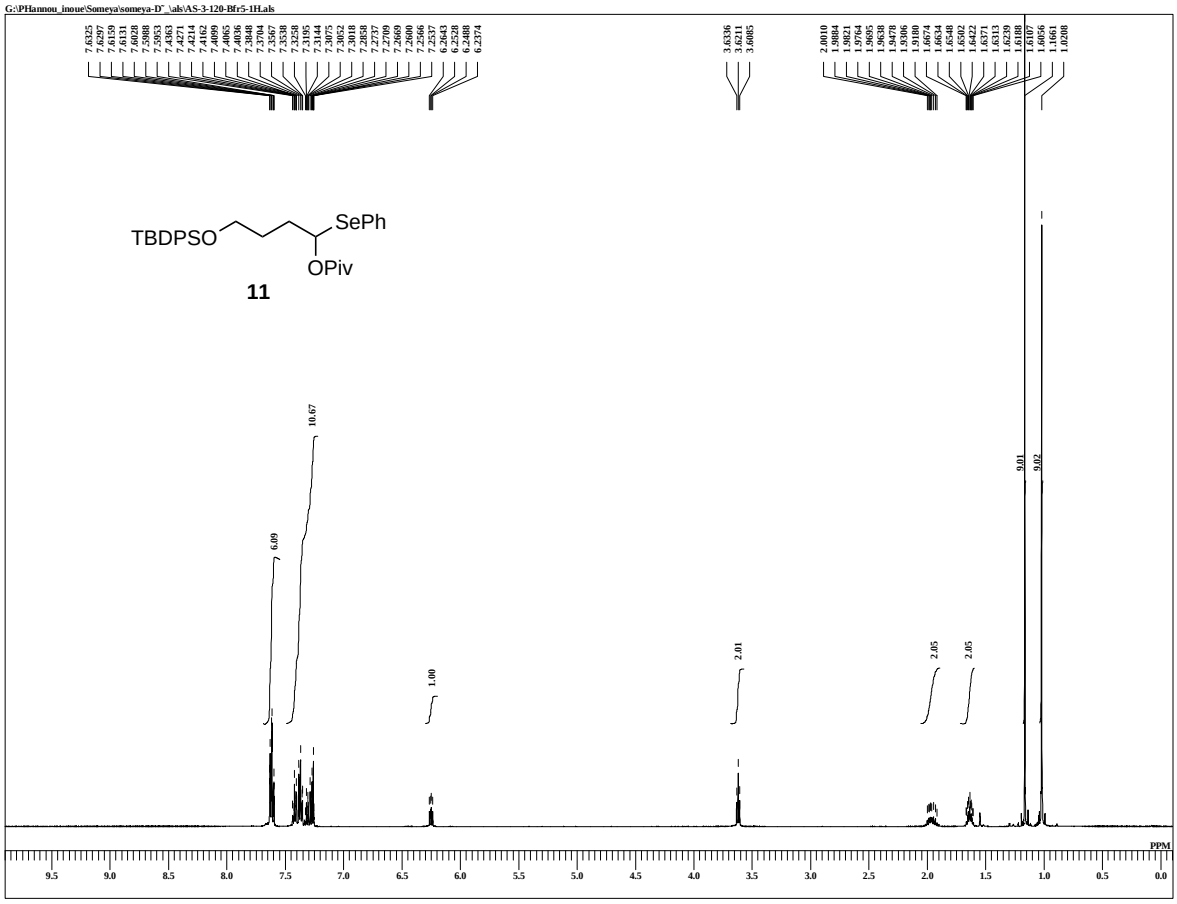
G:\PHannou_inoue\Someya\someya-D^_\als\AS-2-132-fr8-10-13C-1.als




```
DFILE    AS-2-132-fr8-10-13C-1.als
COMNT    AS-2-132-fr8-10-13C
DATIM    26-02-2011 18:29:46
MENUF
OBNUC    13C
OFR      99.55 MHz
OBFRQ    99.55 MHz
OBSET    5.13 KHz
OBFIN    0.98 Hz
PW1      3.33 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    52428
SPO      52428
TIMES    400
DUMMY    4
FREQU    24999.62 Hz
FLT      125000 Hz
DELAY    20.50 usec
ACQTM    1.0486 sec
PD       2.0000 sec
SCANS    400
ADBIT    16
RGAIN    60
BF       1.00 Hz
T1       0.00
T2       0.00
T3       70.00
T4       100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    AS-2-132-fr8-10-13C-1.als
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    20.5 c
SLVNT    CDCL3
EXREF    77.00 ppm
```

AS-3-120-Bfr5-1H

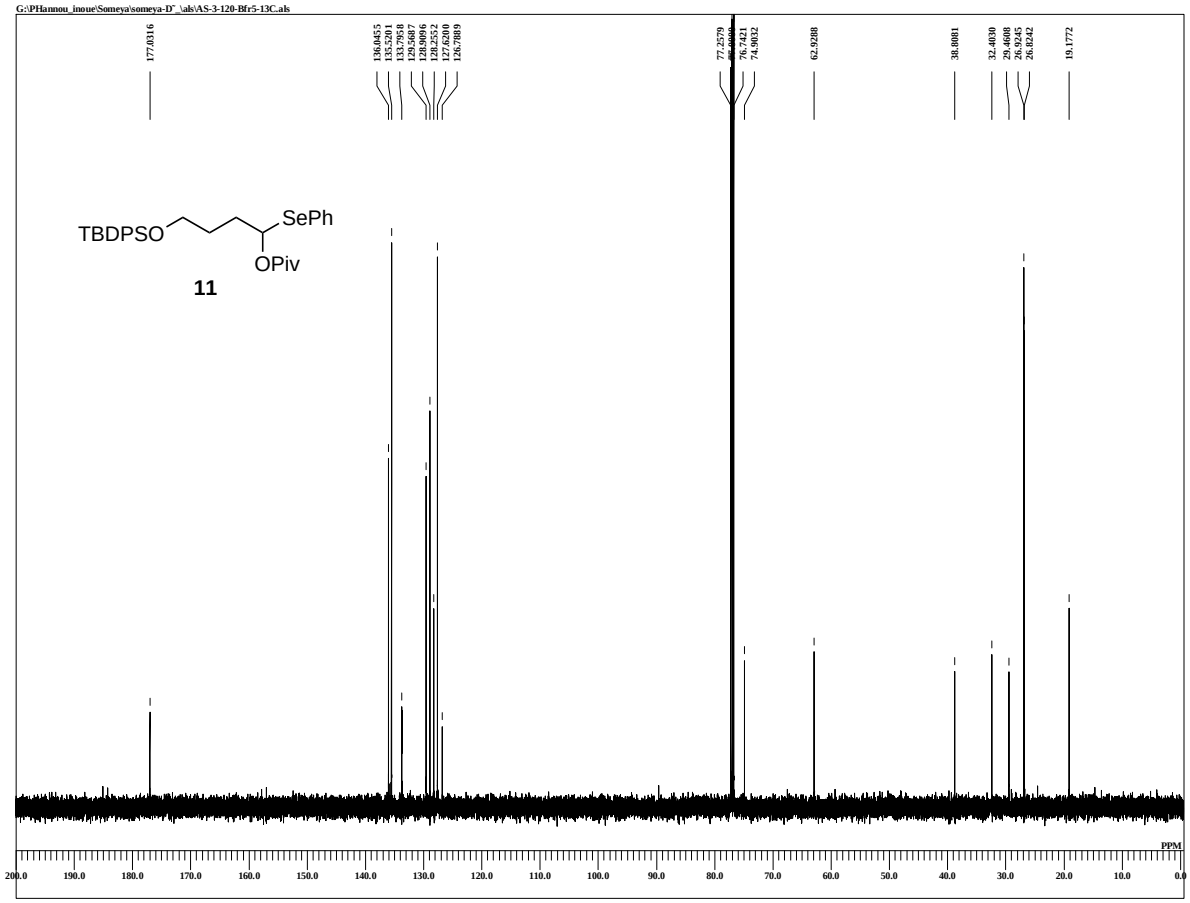
G:\PHannou_inoue\Someya\someya-D^_\als\AS-3-120-Bfr5-1H.als



DFILE AS-3-120-Bfr5-1H.als
COMNT AS-3-120-Bfr5-1H
DATIM 19-10-2009 21:59:42
MENUF
OBNUC 1H
OFR 490.15 MHz
OBFRQ 490.15 MHz
OBSET 9.16 KHz
OBFIN 7.60 Hz
PW1 8.55 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 26214
SPO 26214
TIMES 8
DUMMY 1
FREQU 7352.83 Hz
FLT 37000 Hz
DELAY 13.52 usec
ACQTM 1.7826 sec
PD 5.0000 sec
SCANS 8
ADBIT 16
RGAIN 38
BF 0.01 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 490.15 MHz
IRSET 9.16 KHz
IRFIN 7.60 Hz
IRRPW 92 usec
IRATN 79
DFILE AS-3-120-Bfr5-1H.als
SF
LKSET 70.30 KHz
LKFIN 32.5 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 25.0 c
SLVNT CDCL3
EXREF 7.26 ppm

AS-3-120-Bfr5-13C

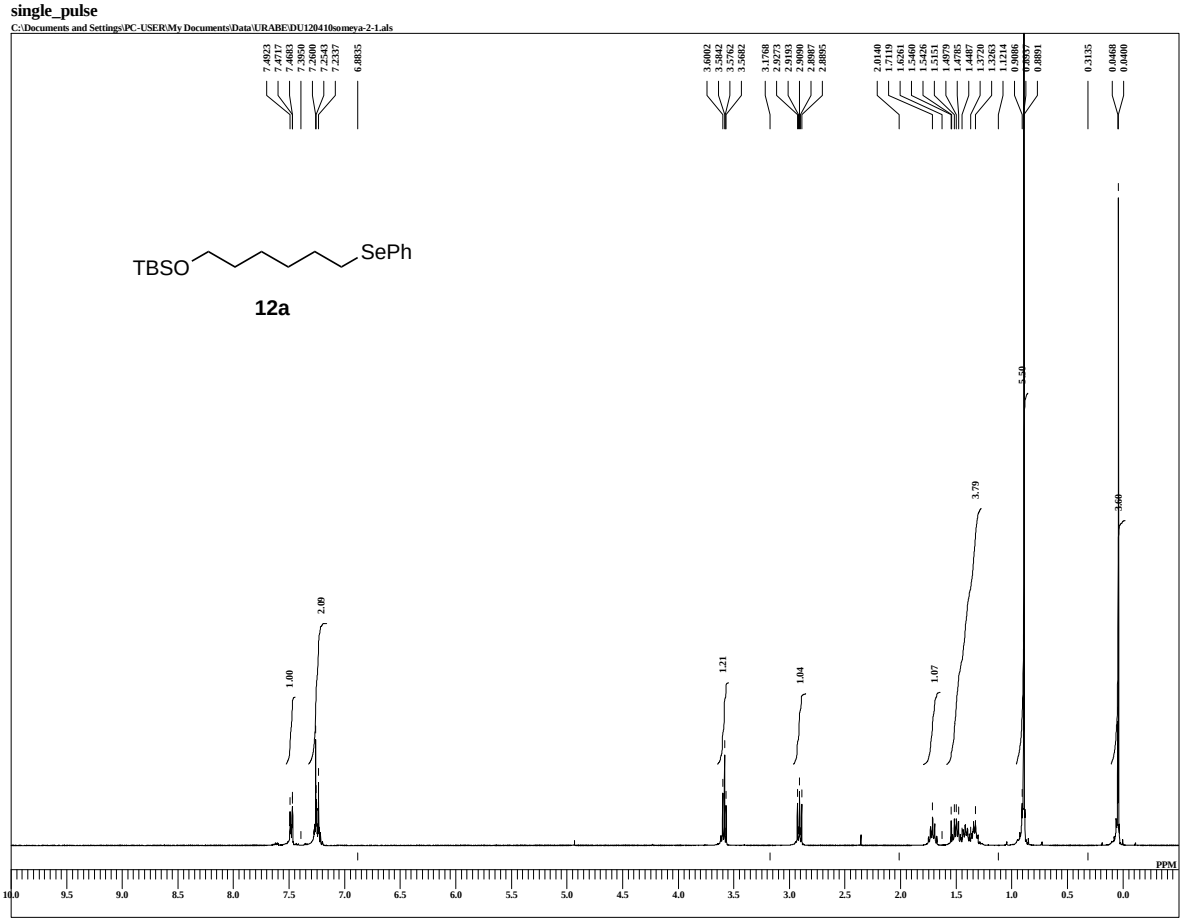
G:\PHannou_inoue\Someya\someya-D^_\als\AS-3-120-Bfr5-13C.als



DFILE AS-3-120-Bfr5-13C.als
COMNT AS-3-120-Bfr5-13C
DATIM 19-10-2009 20:48:41
MENUF
OBNUC 13C
OFR 123.26 MHz
OBFRQ 123.26 MHz
OBSET 2.31 KHz
OBFIN 6.71 Hz
PW1 3.20 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 52428
SPO 52428
TIMES 241
DUMMY 4
FREQU 30863.73 Hz
FLT 155000 Hz
DELAY 21.06 usec
ACQTM 0.8493 sec
PD 2.0000 sec
SCANS 241
ADBIT 16
RGAIN 60
BF 0.01 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 490.15 MHz
IRSET 9.16 KHz
IRFIN 7.60 Hz
IRRPW 92 usec
IRATN 79
DFILE AS-3-120-Bfr5-13C.als
SF
LKSET 70.30 KHz
LKFIN 32.5 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 25.3 c
SLVNT CDCL3
EXREF 77.00 ppm

single_pulse

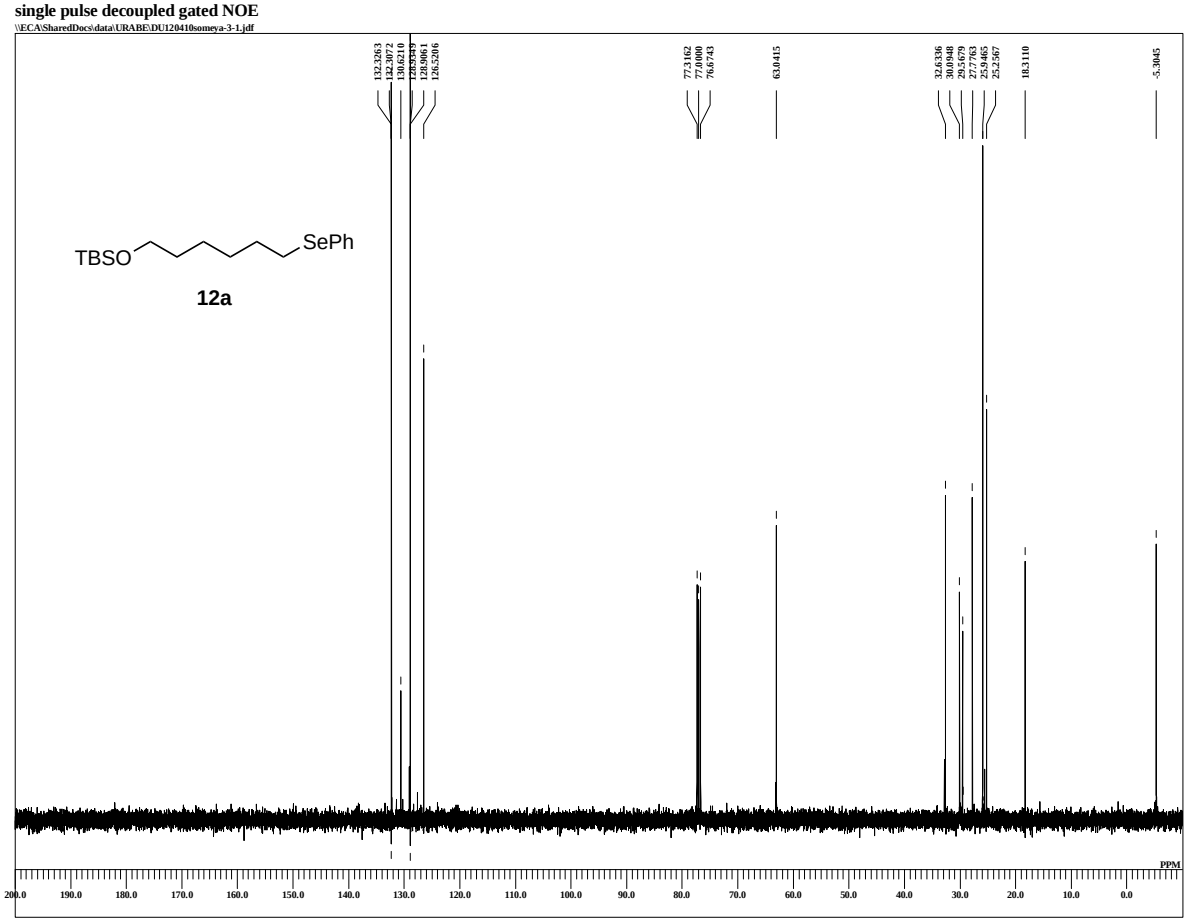
C:\Documents and Settings\PC-USER\My Documents\Data\URABE\DU120410someya-2-1.als



DFILE DU120410someya-2-1.als
COMNT single_pulse
DATIM 10-04-2012 13:26:51
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.62 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 5.0000 sec
SCANS 8
ADBIT 16
RGAIN 40
BF 0.01 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE DU120410someya-2-1.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.3 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

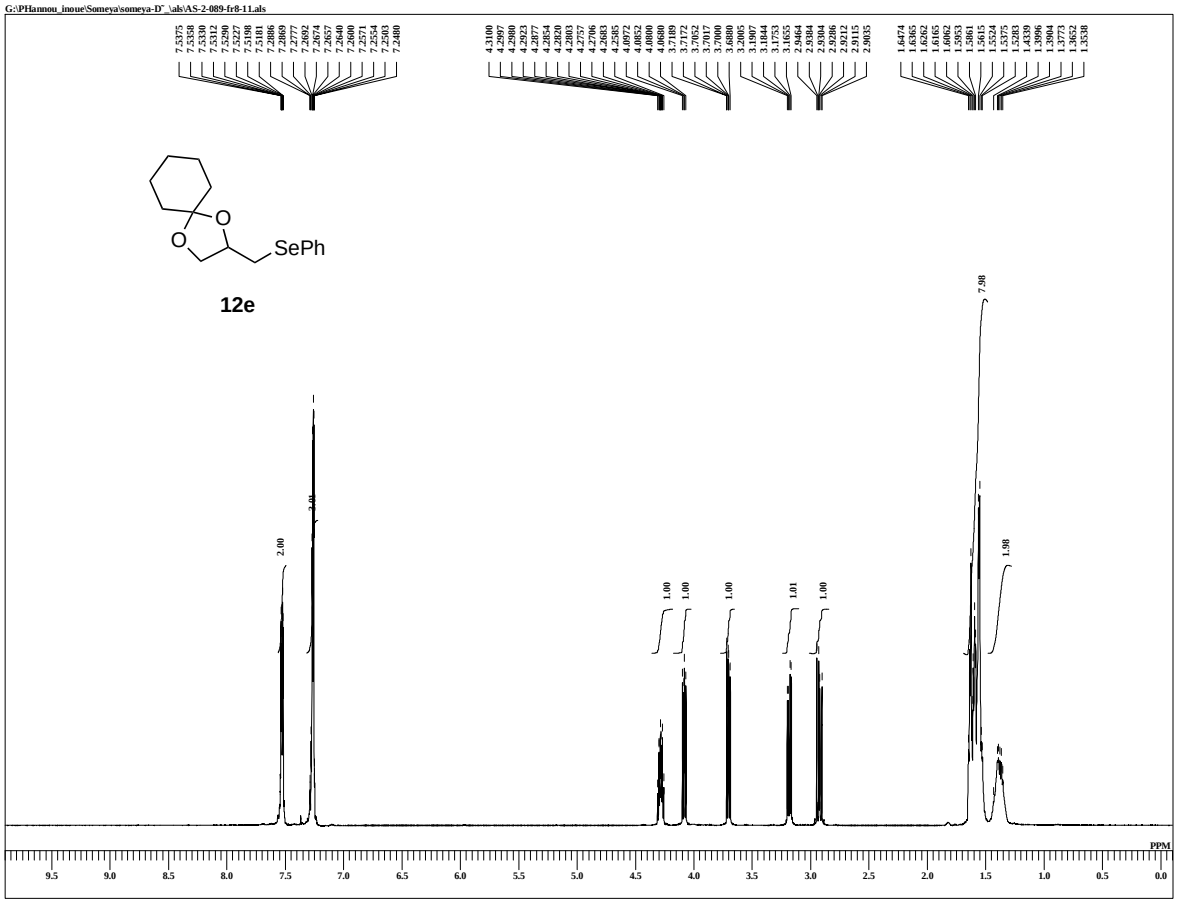
\\ECA\SharedDocs\data\URABE\DU120410someya-3-1.jdf



DFILE DU120410someya-3-1.jdf
COMNT single pulse decoupled gat
DATIM 10-04-2012 13:51:18
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 2.92 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 30
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 30
ADBIT 16
RGAIN 60
BF 0.01 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE DU120410someya-3-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.4 c
SLVNT CDCL3
EXREF 77.00 ppm

AS-2-089-fr8-11

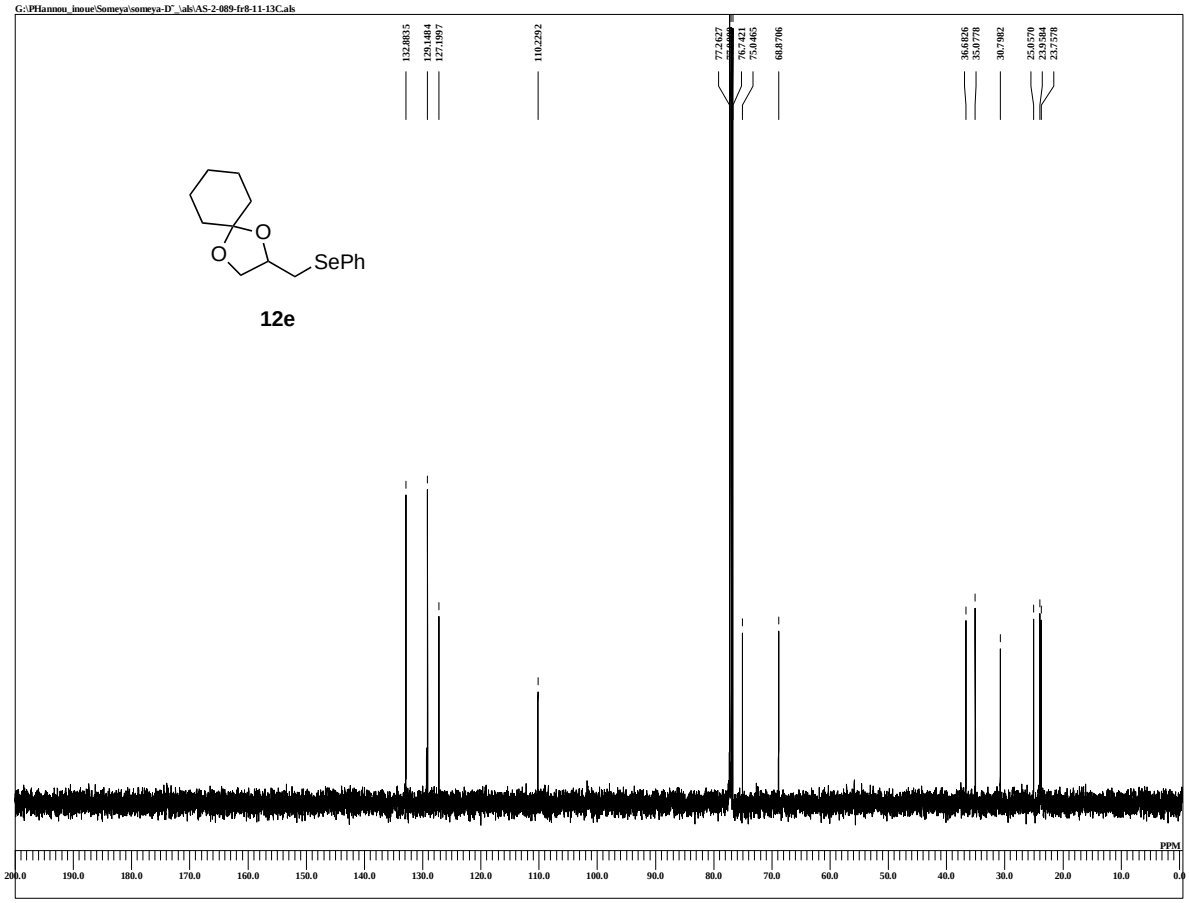
G:\PHannou_inoue\Someya\someya-D~_\als\AS-2-089-fr8-11.als




```
DFILE     AS-2-089-fr8-11.als
COMNT     AS-2-089-fr8-11
DATIM     06-04-2009 14:24:46
MENUF
OBNUC     1H
OFR       490.15 MHz
OBFRQ     490.15 MHz
OBSET     9.16 KHz
OBFIN     7.60 Hz
PW1       8.55 usec
DEADT     0.00 usec
PREDL     0.00000 msec
IWT       1.0000 sec
POINT     26214
SPO       26214
TIMES     8
DUMMY     1
FREQU     7352.83 Hz
FLT       37000 Hz
DELAY     13.52 usec
ACQTM     1.7826 sec
PD        2.0000 sec
SCANS     8
ADBIT     16
RGAIN     36
BF        0.01 Hz
T1        0.00
T2        0.00
T3        70.00
T4        100.00
EXMOD     single_pulse.ex2
EXPCM
IRNUC     1H
IFR       490.15 MHz
IRSET     9.16 KHz
IRFIN     7.60 Hz
IRRPW     92 usec
IRATN     79
DFILE     AS-2-089-fr8-11.als
SF
LKSET     70.30 KHz
LKFIN     32.5 Hz
LKLEV     0
LGAIN     0
LKPHS     0
LKSIG     0
CSPED     0 Hz
FILDC
FILDF
CTEMP     18.3 c
SLVNT     CDCL3
EXREF     7.26 ppm
```

AS-2-089-fr8-11-13C

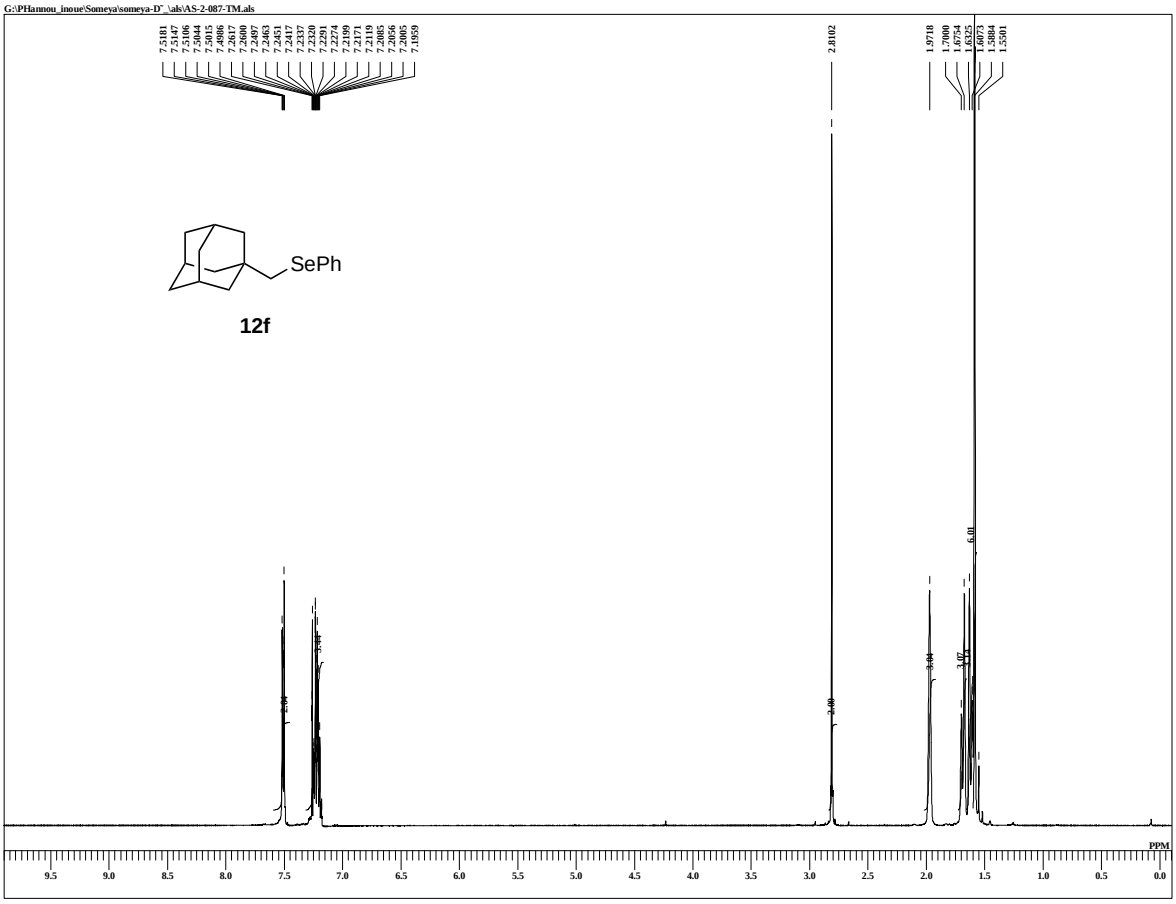
G:\PHannou_inoue\Someya\someya-D~_\als\AS-2-089-fr8-11-13C.als




```
DFILE     AS-2-089-fr8-11-13C.als
COMNT     AS-2-089-fr8-11-13C
DATIM     06-04-2009 14:26:59
MENUF
OBNUC     13C
OFR       123.26 MHz
OBFRQ     123.26 MHz
OBSET     2.31 KHz
OBFIN     6.71 Hz
PW1       3.20 usec
DEADT     0.00 usec
PREDL     0.00000 msec
IWT       1.0000 sec
POINT     52428
SPO       52428
TIMES     41
DUMMY     4
FREQU     30863.73 Hz
FLT       155000 Hz
DELAY     21.06 usec
ACQTM     0.8493 sec
PD        2.0000 sec
SCANS     41
ADBIT     16
RGAIN     50
BF        1.00 Hz
T1        0.00
T2        0.00
T3        70.00
T4        100.00
EXMOD     single_pulse_dec
EXPCM
IRNUC     1H
IFR       490.15 MHz
IRSET     9.16 KHz
IRFIN     7.60 Hz
IRRPW     92 usec
IRATN     79
DFILE     AS-2-089-fr8-11-13C.als
SF
LKSET     70.30 KHz
LKFIN     32.5 Hz
LKLEV     0
LGAIN     0
LKPHS     0
LKSIG     0
CSPED     0 Hz
FILDC
FILDF
CTEMP     18.6 c
SLVNT     CDCL3
EXREF     77.00 ppm
```

AS-2-087-TM

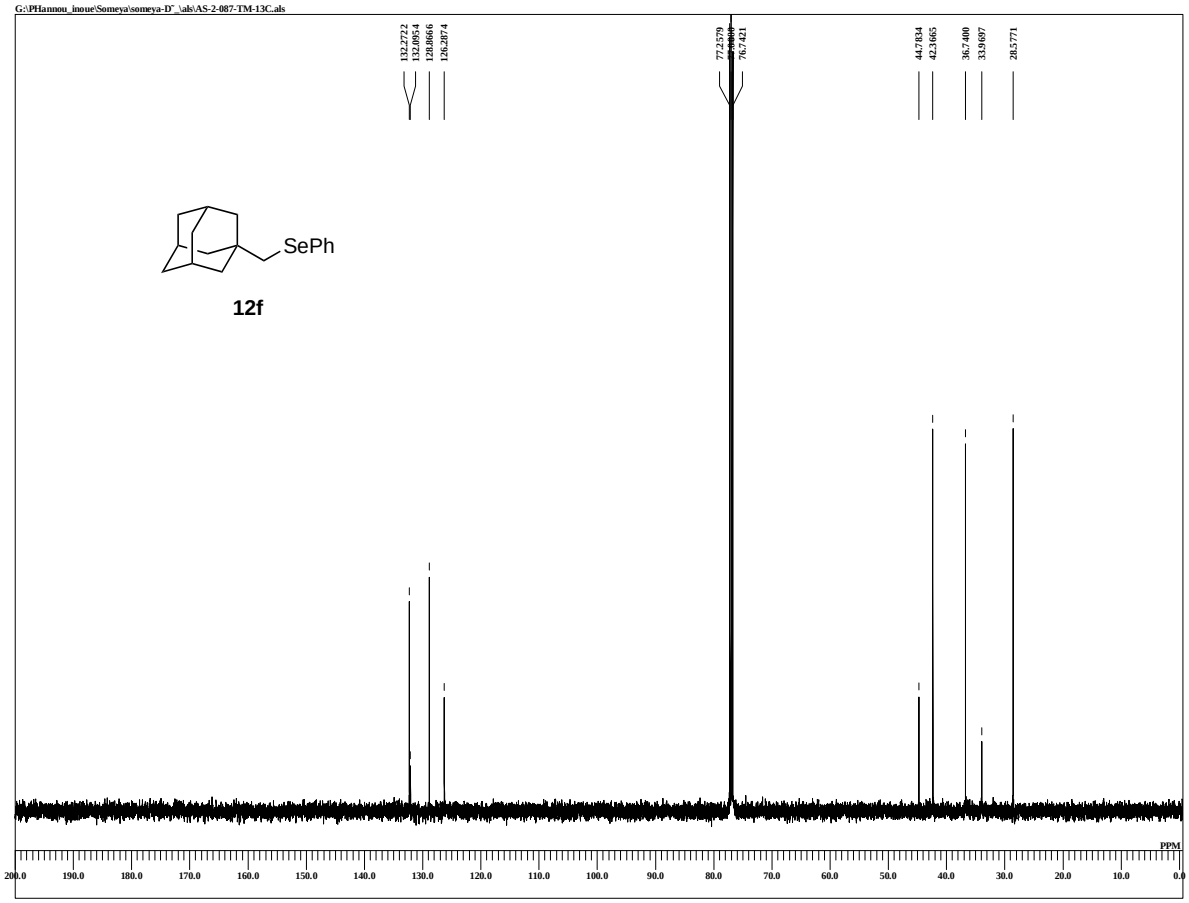
G:\PHannou_inoue\Someya\someya-D~_\als\AS-2-087-TM.als



DFILE AS-2-087-TM.als
COMNT AS-2-087-TM
DATIM 24-04-2009 11:44:44
MENUF
OBNUC 1H
OFR 490.15 MHz
OBFRQ 490.15 MHz
OBSET 9.16 KHz
OBFIN 7.60 Hz
PW1 8.55 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 26214
SPO 26214
TIMES 8
DUMMY 1
FREQU 7352.83 Hz
FLT 37000 Hz
DELAY 13.52 usec
ACQTM 1.7826 sec
PD 5.0000 sec
SCANS 8
ADBIT 16
RGAIN 40
BF 0.01 Hz
T1 0.00
T2 0.00
T3 70.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 490.15 MHz
IRSET 9.16 KHz
IRFIN 7.60 Hz
IRRPW 92 usec
IRATN 79
DFILE AS-2-087-TM.als
SF
LKSET 70.30 KHz
LKFIN 32.5 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.2 c
SLVNT CDCL3
EXREF 7.26 ppm

AS-2-061-fr5-6-13C

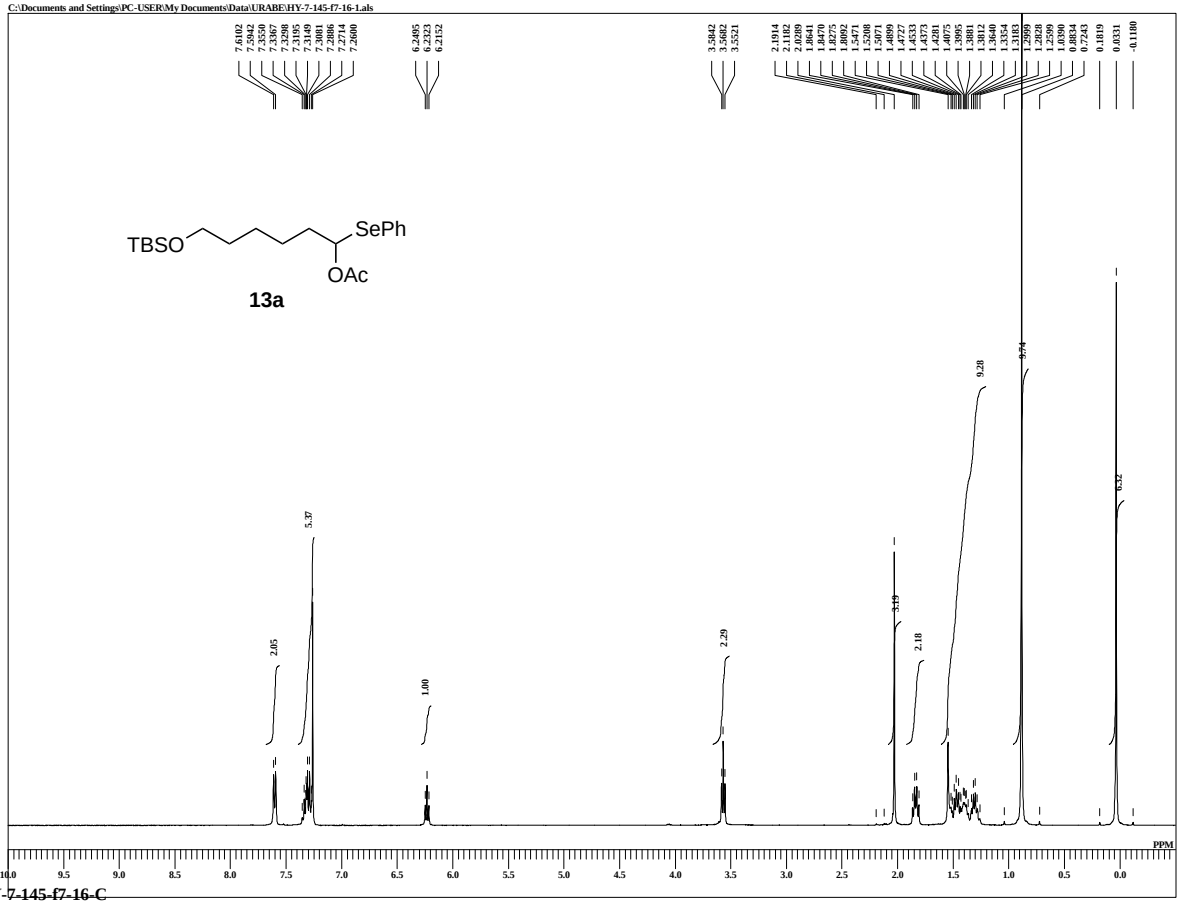
G:\PHannou_inoue\Someya\someya-D~_\als\AS-2-087-TM-13C.als



DFILE AS-2-087-TM-13C.als
COMNT AS-2-061-fr5-6-13C
DATIM 24-04-2009 11:52:24
MENUF
OBNUC 13C
OFR 123.26 MHz
OBFRQ 123.26 MHz
OBSET 2.31 KHz
OBFIN 6.71 Hz
PW1 3.20 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 52428
SPO 52428
TIMES 141
DUMMY 4
FREQU 30863.73 Hz
FLT 155000 Hz
DELAY 21.06 usec
ACQTM 0.8493 sec
PD 2.0000 sec
SCANS 141
ADBIT 16
RGAIN 50
BF 1.00 Hz
T1 0.00
T2 0.00
T3 70.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 490.15 MHz
IRSET 9.16 KHz
IRFIN 7.60 Hz
IRRPW 92 usec
IRATN 79
DFILE AS-2-087-TM-13C.als
SF
LKSET 70.30 KHz
LKFIN 32.5 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.7 c
SLVNT CDCL3
EXREF 77.00 ppm

HY-7-145-f7-16

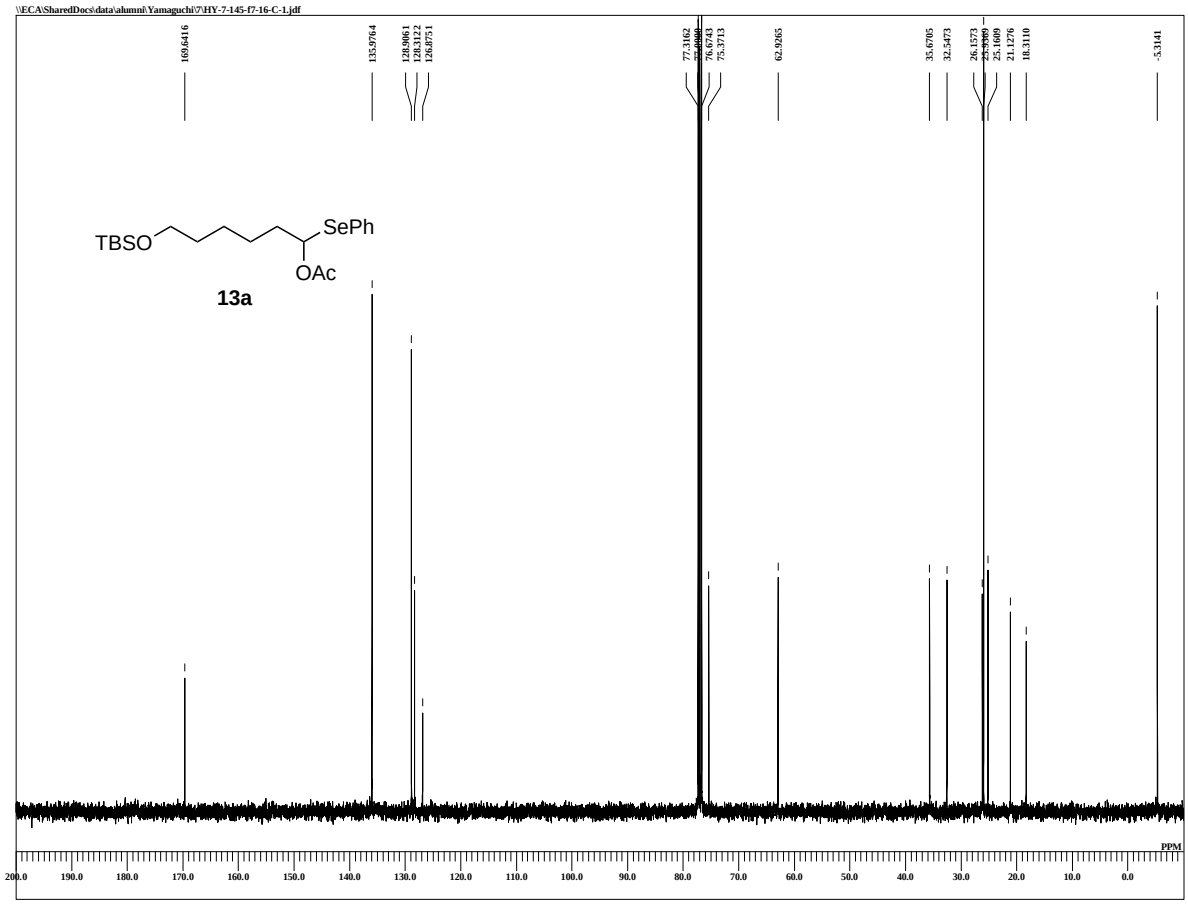
C:\Documents and Settings\PC-USER\My Documents\Data\URABE\HY-7-145-f7-16-1.als



DFILE HY-7-145-f7-16-1.als
COMNT HY-7-145-f7-16
DATIM 29-09-2011 22:36:20
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.62 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 5.0000 sec
SCANS 8
ADBIT 16
RGAIN 48
BF 1.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE HY-7-145-f7-16-1.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 24.8 c
SLVNT CDCL3
EXREF 7.26 ppm

HY-7-145-f7-16-C

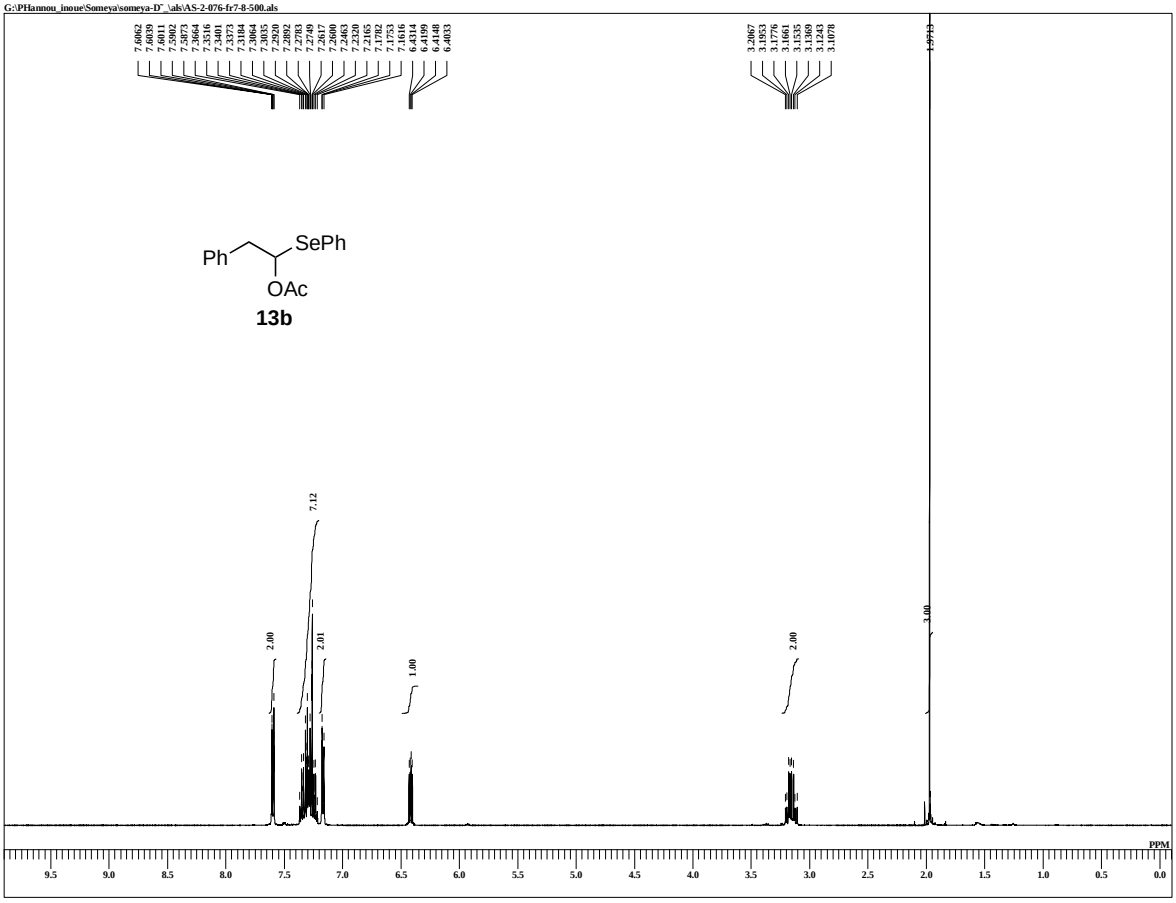
\\ECA\SharedDocs\data\alumni\Yamaguchi\7\HY-7-145-f7-16-C-1.jdf



DFILE HY-7-145-f7-16-C-1.jdf
COMNT HY-7-145-f7-16-C
DATIM 29-09-2011 22:50:50
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 2.92 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 52
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 8.0000 sec
SCANS 52
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE HY-7-145-f7-16-C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 25.2 c
SLVNT CDCL3
EXREF 77.00 ppm

AS-2-076-fr7-8-500

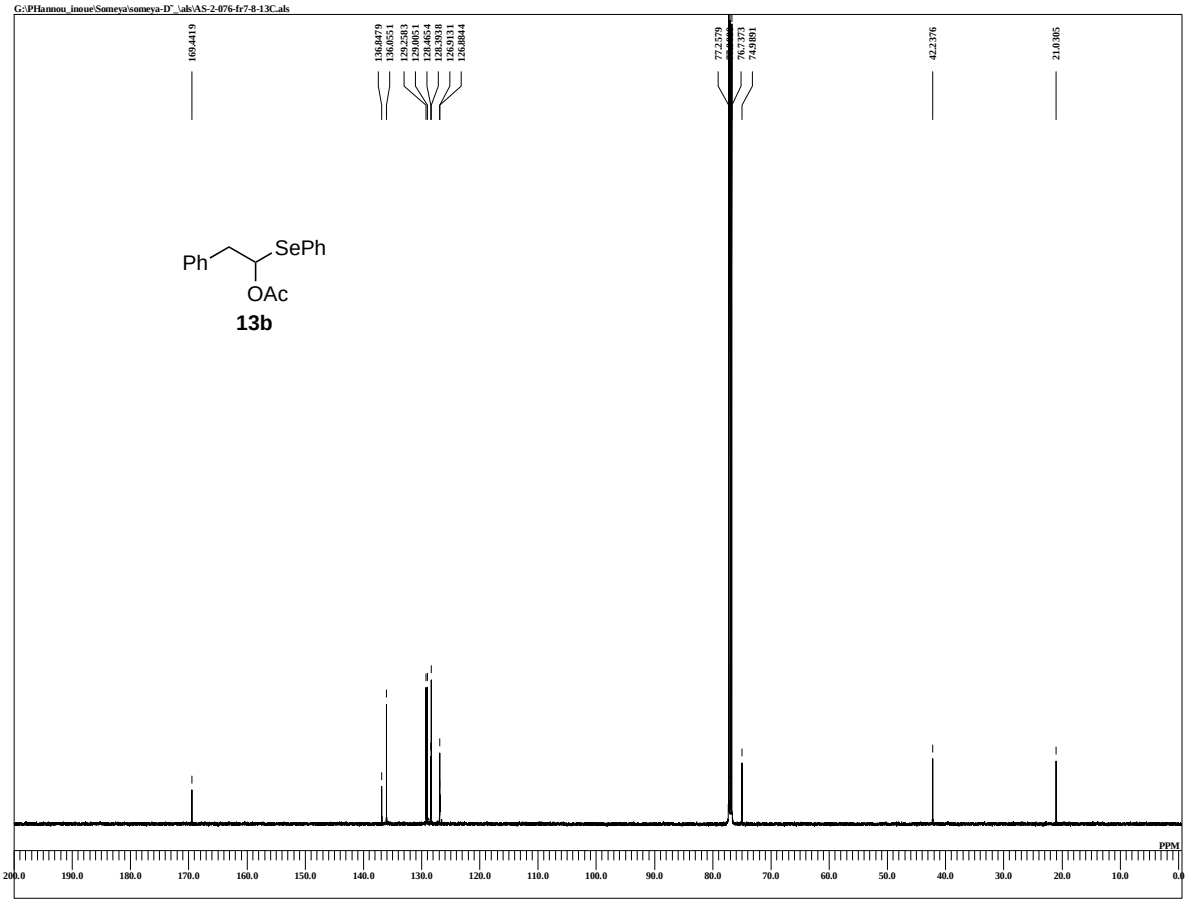
G:\PHannou_inoue\Someya\someya-D~_\als\AS-2-076-fr7-8-500.als




```
DFILE    AS-2-076-fr7-8-500.als
COMNT    AS-2-076-fr7-8-500
DATIM    26-04-2009 18:08:31
MENUF
OBNUC    1H
OFR      490.15 MHz
OBFRQ    490.15 MHz
OBSET    9.16 KHz
OBFIN    7.60 Hz
PW1      8.55 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    26214
SPO      26214
TIMES    8
DUMMY    1
FREQU    7352.83 Hz
FLT      37000 Hz
DELAY    13.52 usec
ACQTM    1.7826 sec
PD       5.0000 sec
SCANS    8
ADBIT    16
RGAIN    44
BF       0.01 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR      490.15 MHz
IRSET    9.16 KHz
IRFIN    7.60 Hz
IRRPW    118 usec
IRATN    79
DFILE    AS-2-076-fr7-8-500.als
SF
LKSET    70.30 KHz
LKFIN    32.5 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    19.9 c
SLVNT    CDCL3
EXREF    7.26 ppm
```

AS-2-076-fr7-8-13C

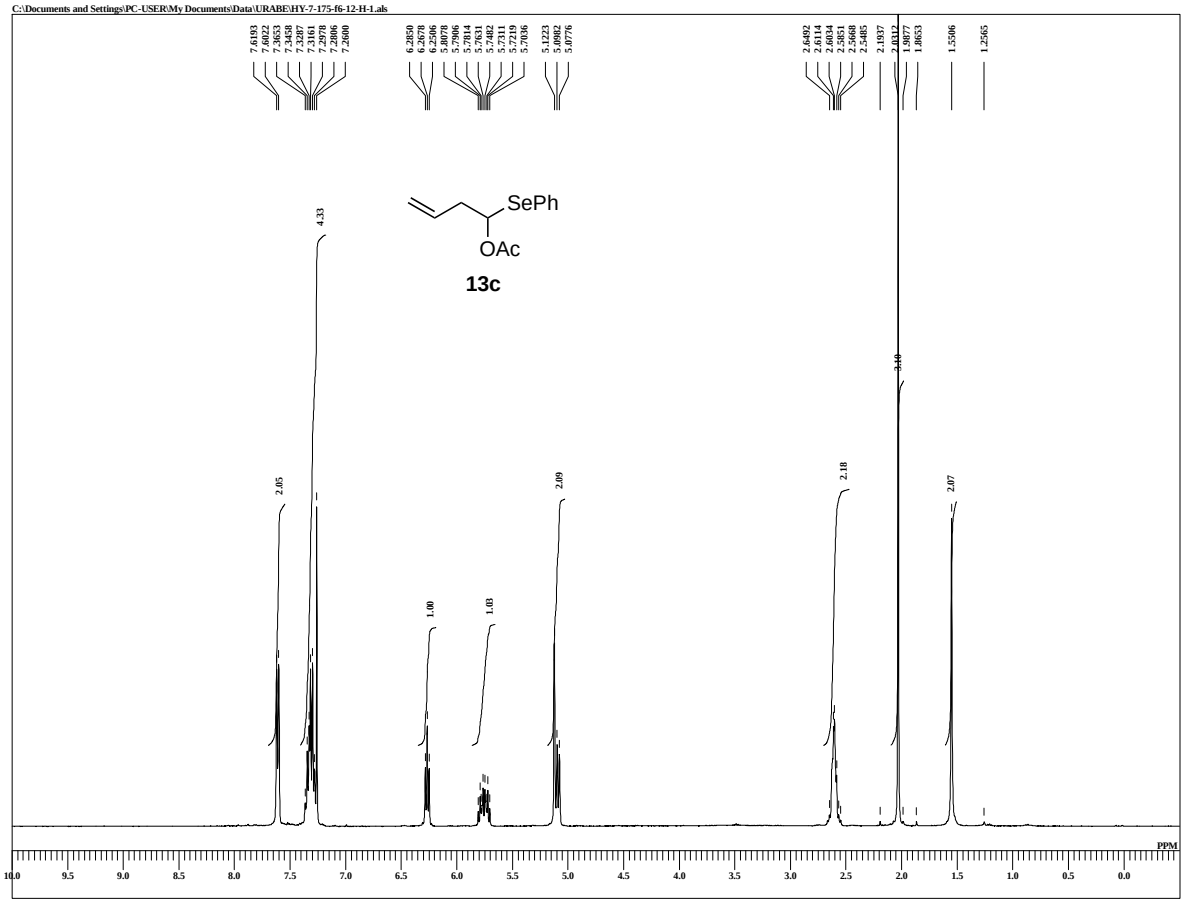
G:\PHannou_inoue\Someya\someya-D~_\als\AS-2-076-fr7-8-13C.als




```
DFILE    AS-2-076-fr7-8-13C..als
COMNT    AS-2-076-fr7-8-13C
DATIM    26-04-2009 21:56:45
MENUF
OBNUC    13C
OFR      123.26 MHz
OBFRQ    123.26 MHz
OBSET    2.31 KHz
OBFIN    6.71 Hz
PW1      3.20 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    52428
SPO      52428
TIMES    4800
DUMMY    4
FREQU    30863.73 Hz
FLT      155000 Hz
DELAY    21.06 usec
ACQTM    0.8493 sec
PD       2.0000 sec
SCANS    4800
ADBIT    16
RGAIN    50
BF       1.00 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR      490.15 MHz
IRSET    9.16 KHz
IRFIN    7.60 Hz
IRRPW    92 usec
IRATN    79
DFILE    AS-2-076-fr7-8-13C..als
SF
LKSET    70.30 KHz
LKFIN    32.5 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    20.9 c
SLVNT    CDCL3
EXREF    77.00 ppm
```

HY-7-175-f6-12-H

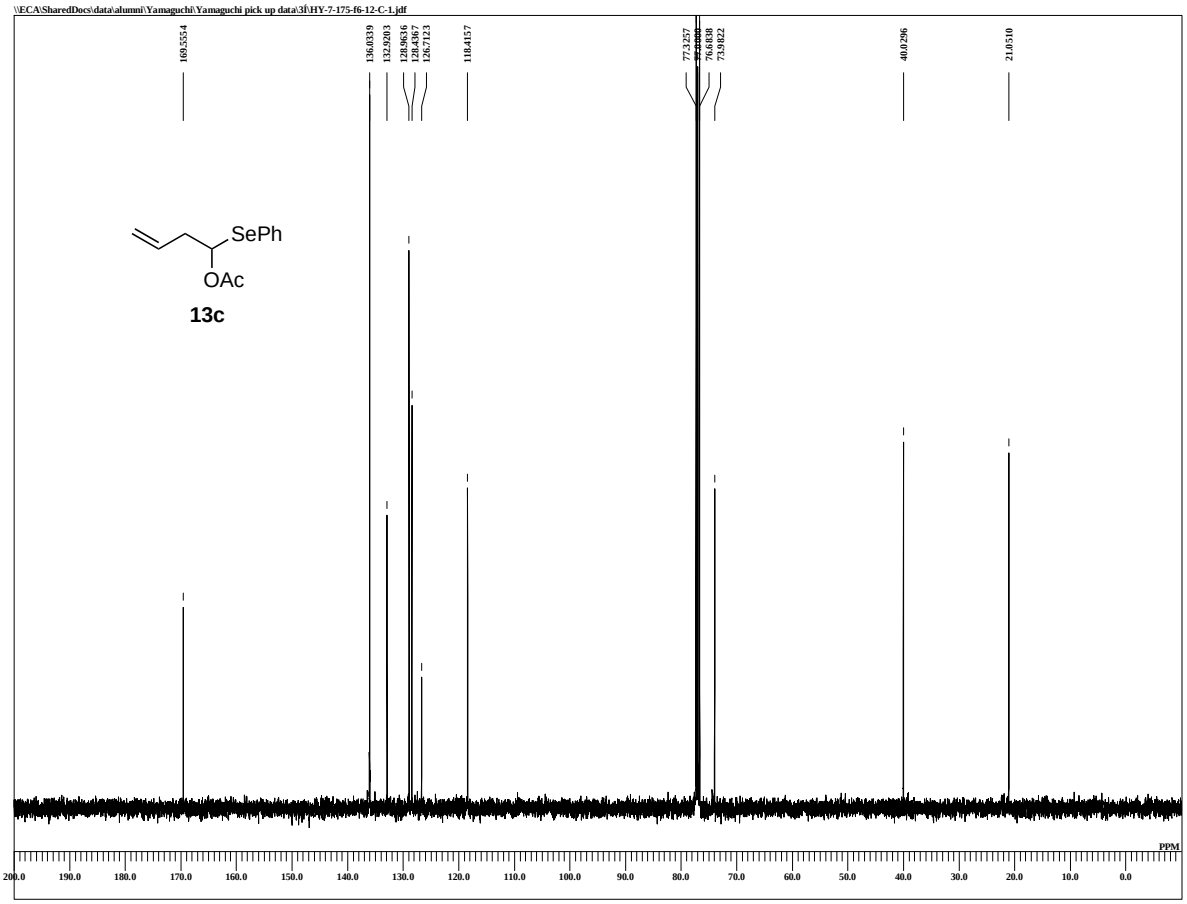
C:\Documents and Settings\PC-USER\My Documents\Data\URABE\HY-7-175-f6-12-H-1.als




```
DFILE     HY-7-175-f6-12-H-1.als
COMNT     HY-7-175-f6-12-H
DATIM     29-10-2011 15:27:35
MENUF
OBNUC     1H
OFR       395.88 MHz
OBFRQ     395.88 MHz
OBSET     6.28 KHz
OBFIN     0.87 Hz
PW1       6.62 usec
DEADT     0.00 usec
PREDL     0.00000 msec
IWT       1.0000 sec
POINT     13107
SPO       13107
TIMES     8
DUMMY     1
FREQU     5938.15 Hz
FLT       30000 Hz
DELAY     16.68 usec
ACQTM     2.2073 sec
PD        5.0000 sec
SCANS     8
ADBIT     16
RGAIN     48
BF        1.00 Hz
T1        0.00
T2        0.00
T3        90.00
T4        100.00
EXMOD     single_pulse.ex2
EXPCM
IRNUC     1H
IFR       395.88 MHz
IRSET     6.28 KHz
IRFIN     0.87 Hz
IRRPW     115 usec
IRATN     79
DFILE     HY-7-175-f6-12-H-1.als
SF
LKSET     13.20 KHz
LKFIN     75.7 Hz
LKLEV     0
LGAIN     0
LKPHS     0
LKSIG     0
CSPED     0 Hz
FILDC
FILDF
CTEMP     25.2 c
SLVNT     CDCL3
EXREF     7.26 ppm
```

HY-7-175-f6-12-C

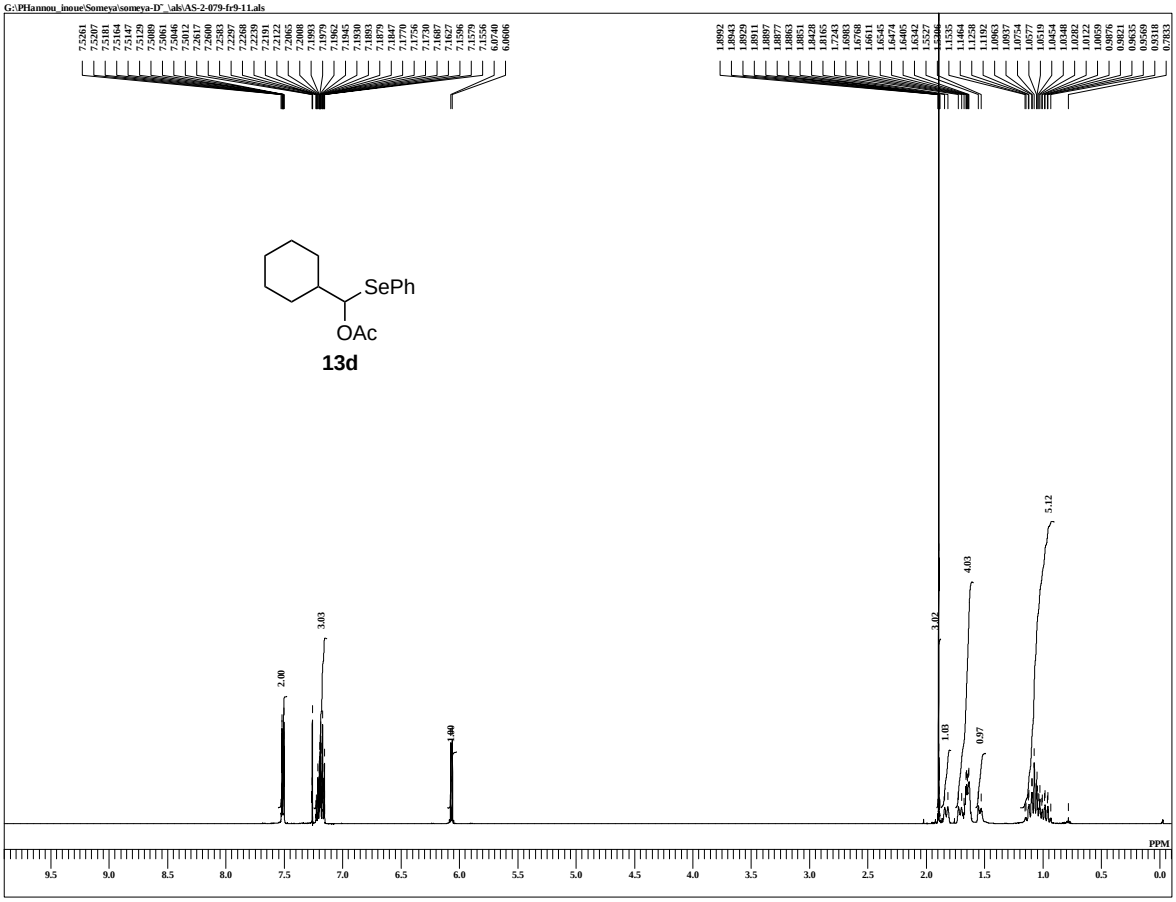
\\ECA\SharedDocs\data\alumni\Yamaguchi\Yamaguchi pick up data\3月\HY-7-175-f6-12-C-1.jdf




```
DFILE     HY-7-175-f6-12-C-1.jdf
COMNT     HY-7-175-f6-12-C
DATIM     29-10-2011 15:38:56
MENUF
OBNUC     13C
OFR       99.55 MHz
OBFRQ     99.55 MHz
OBSET     5.13 KHz
OBFIN     0.98 Hz
PW1       2.92 usec
DEADT     0.00 usec
PREDL     0.00000 msec
IWT       1.0000 sec
POINT     32768
SPO       32768
TIMES     36
DUMMY     4
FREQU     31250.00 Hz
FLT       125000 Hz
DELAY     20.50 usec
ACQTM     1.0486 sec
PD        8.0000 sec
SCANS     36
ADBIT     16
RGAIN     60
BF        1.00 Hz
T1        0.00
T2        0.00
T3        90.00
T4        100.00
EXMOD     single_pulse_dec
EXPCM
IRNUC     1H
IFR       395.88 MHz
IRSET     6.28 KHz
IRFIN     0.87 Hz
IRRPW     115 usec
IRATN     79
DFILE     HY-7-175-f6-12-C-1.jdf
SF
LKSET     13.20 KHz
LKFIN     75.7 Hz
LKLEV     0
LGAIN     0
LKPHS     0
LKSIG     0
CSPED     0 Hz
FILDC
FILDF
CTEMP     25.3 c
SLVNT     CDCL3
EXREF     77.00 ppm
```

AS-2-079-fr9-11

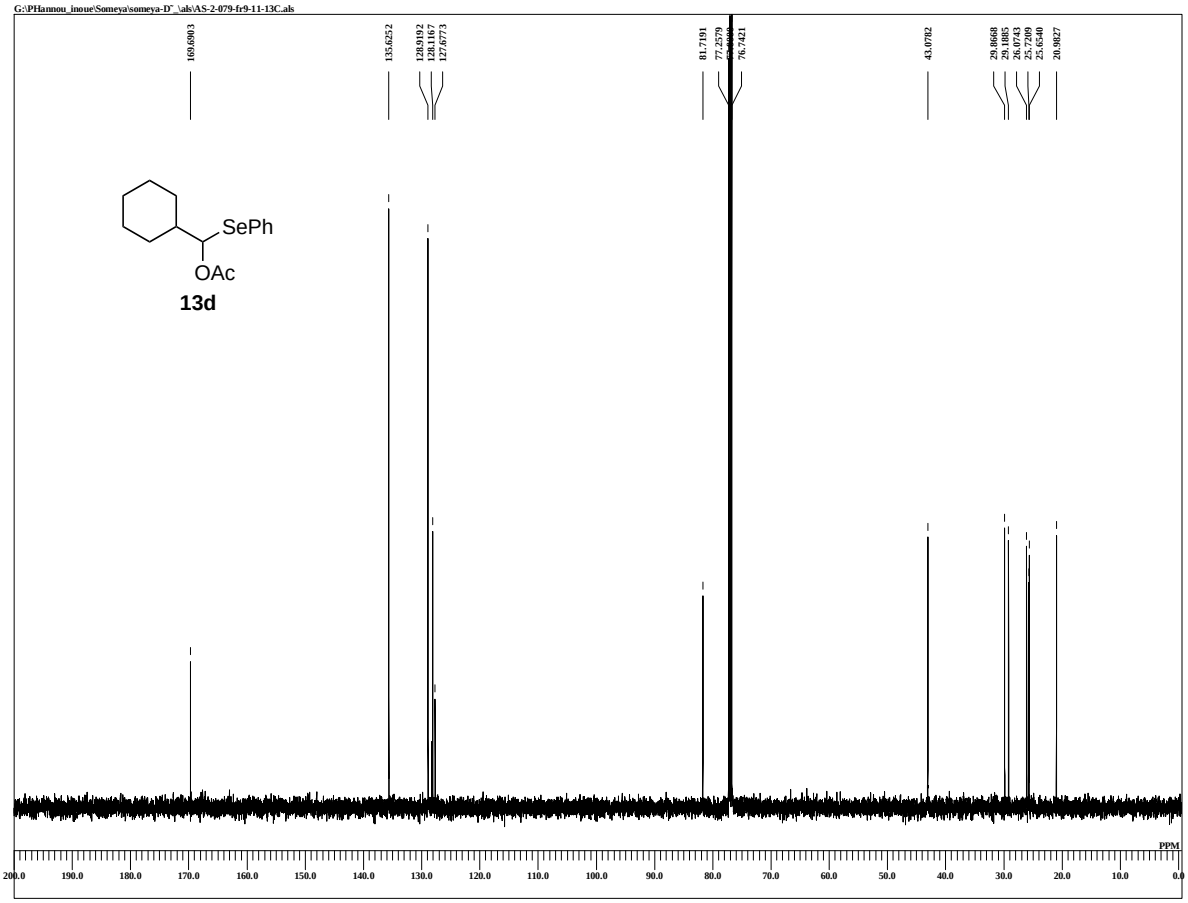
G:\PHannou_inoue\Someya\someya-D~_\als\AS-2-079-fr9-11.als



DFILE AS-2-079-fr9-11.als
COMNT AS-2-079-fr9-11
DATIM 24-04-2009 12:48:28
MENUF
OBNUC 1H
OFR 490.15 MHz
OBFRQ 490.15 MHz
OBSET 9.16 KHz
OBFIN 7.60 Hz
PW1 8.55 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 52428
SPO 52428
TIMES 8
DUMMY 1
FREQU 7352.83 Hz
FLT 37000 Hz
DELAY 13.52 usec
ACQTM 1.7826 sec
PD 5.0000 sec
SCANS 8
ADBIT 16
RGAIN 34
BF 0.01 Hz
T1 0.00
T2 0.00
T3 70.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 490.15 MHz
IRSET 9.16 KHz
IRFIN 7.60 Hz
IRRPW 92 usec
IRATN 79
DFILE AS-2-079-fr9-11.als
SF
LKSET 70.30 KHz
LKFIN 32.5 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.6 c
SLVNT CDCL3
EXREF 7.26 ppm

AS-2-079-fr9-11-13C

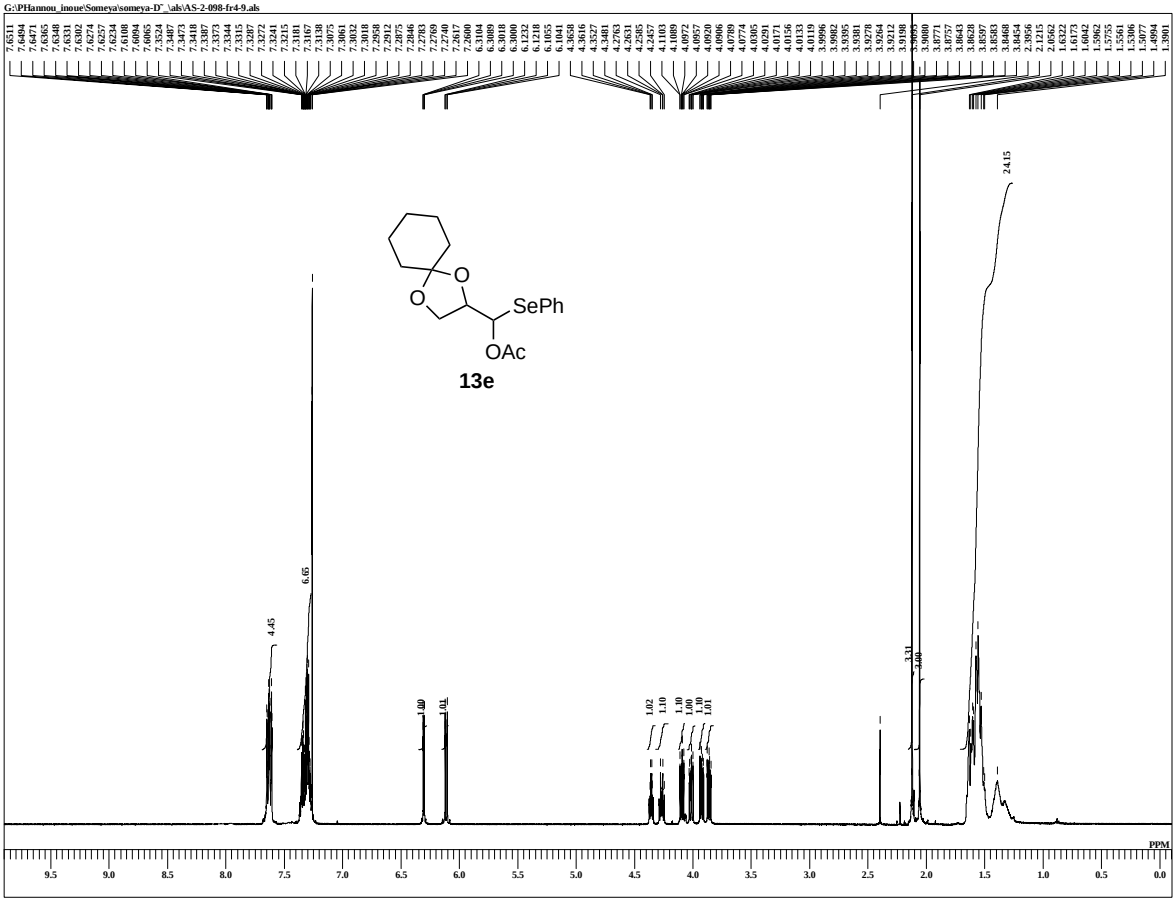
G:\PHannou_inoue\Someya\someya-D~_\als\AS-2-079-fr9-11-13C.als



DFILE AS-2-079-fr9-11-13C.als
COMNT AS-2-079-fr9-11-13C
DATIM 24-04-2009 12:52:58
MENUF
OBNUC 13C
OFR 123.26 MHz
OBFRQ 123.26 MHz
OBSET 2.31 KHz
OBFIN 6.71 Hz
PW1 3.20 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 26214
SPO 26214
TIMES 89
DUMMY 4
FREQU 30863.73 Hz
FLT 155000 Hz
DELAY 21.06 usec
ACQTM 0.8493 sec
PD 2.0000 sec
SCANS 89
ADBIT 16
RGAIN 50
BF 1.00 Hz
T1 0.00
T2 0.00
T3 70.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 490.15 MHz
IRSET 9.16 KHz
IRFIN 7.60 Hz
IRRPW 92 usec
IRATN 79
DFILE AS-2-079-fr9-11-13C.als
SF
LKSET 70.30 KHz
LKFIN 32.5 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.9 c
SLVNT CDCL3
EXREF 77.00 ppm

AS-2-098-fr4-9

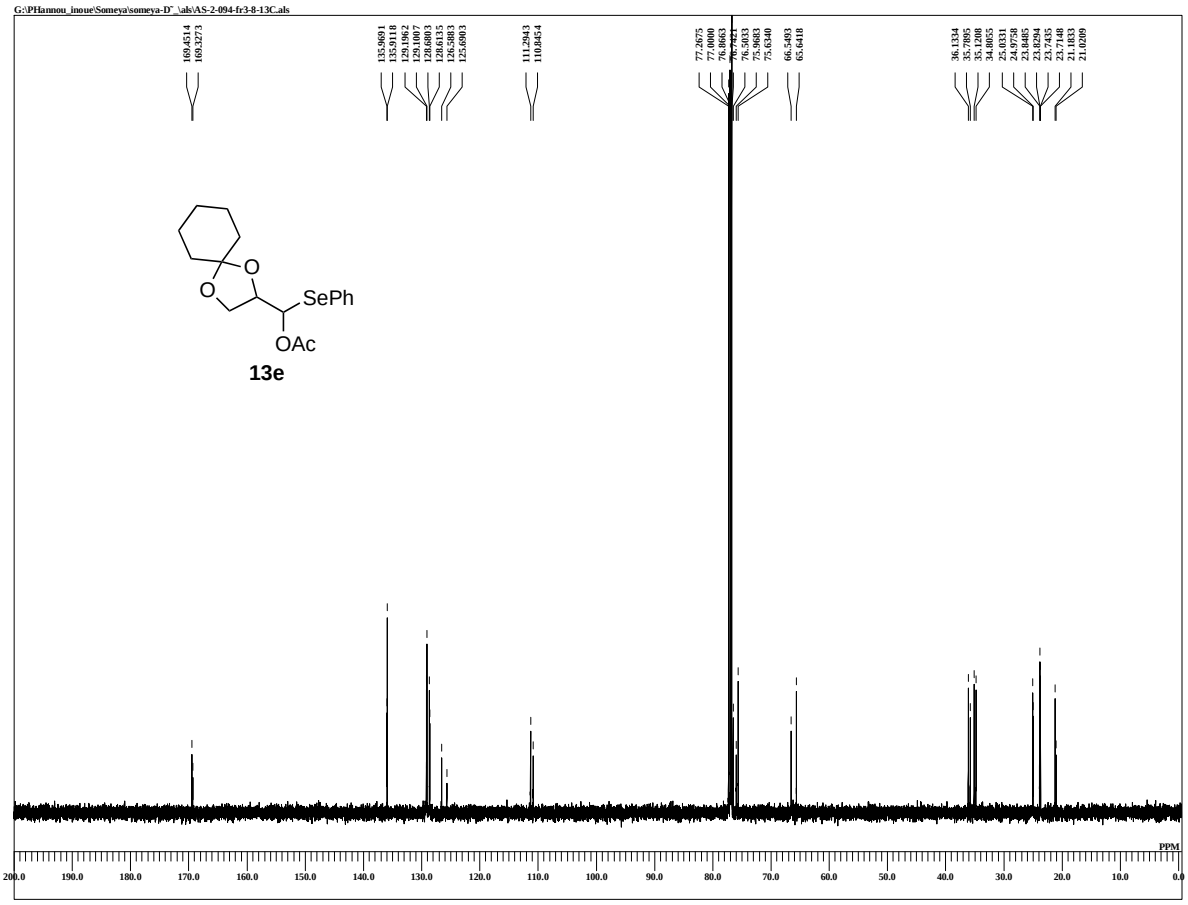
G:\PHannou_inoue\Someya\someya-D^_\als\AS-2-098-fr4-9.als



DFILE AS-2-098-fr4-9.als
COMNT AS-2-098-fr4-9
DATIM 10-04-2009 14:15:07
MENUF
OBNUC 1H
OFR 490.15 MHz
OBFRQ 490.15 MHz
OBSET 9.16 KHz
OBFIN 7.60 Hz
PW1 8.55 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 52428
SPO 52428
TIMES 8
DUMMY 1
FREQU 7352.83 Hz
FLT 37000 Hz
DELAY 13.52 usec
ACQTM 1.7826 sec
PD 5.0000 sec
SCANS 8
ADBIT 16
RGAIN 44
BF 0.01 Hz
T1 0.00
T2 0.00
T3 70.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 490.15 MHz
IRSET 9.16 KHz
IRFIN 7.60 Hz
IRRPW 92 usec
IRATN 79
DFILE AS-2-098-fr4-9.als
SF
LKSET 70.30 KHz
LKFIN 32.5 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 19.5 c
SLVNT CDCL3
EXREF 7.26 ppm

AS-2-094-fr3-8-13C

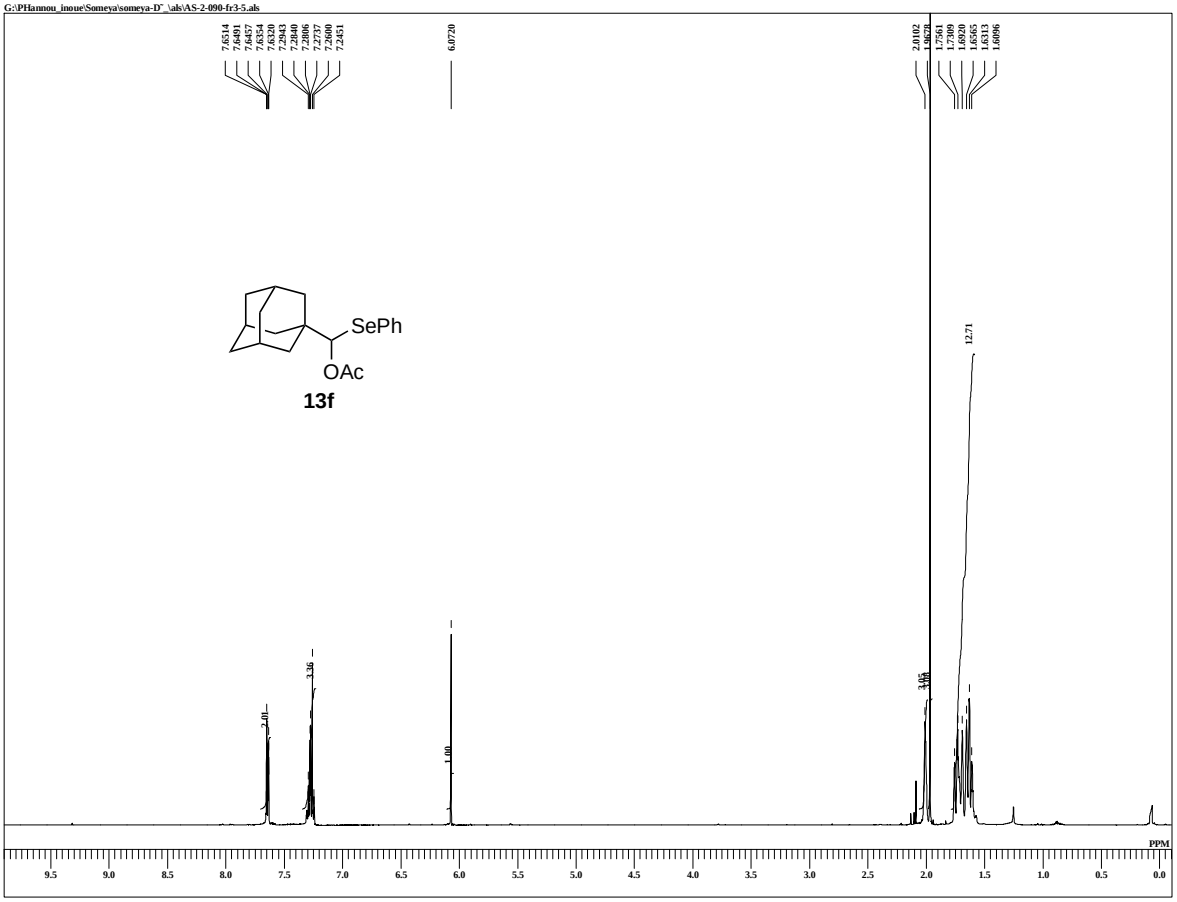
G:\PHannou_inoue\Someya\someya-D^_\als\AS-2-094-fr3-8-13C.als



DFILE AS-2-094-fr3-8-13C.als
COMNT AS-2-094-fr3-8-13C
DATIM 07-04-2009 23:12:28
MENUF
OBNUC 13C
OFR 123.26 MHz
OBFRQ 123.26 MHz
OBSET 2.31 KHz
OBFIN 6.71 Hz
PW1 3.20 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 26214
SPO 26214
TIMES 1000
DUMMY 4
FREQU 30863.73 Hz
FLT 155000 Hz
DELAY 21.06 usec
ACQTM 0.8493 sec
PD 2.0000 sec
SCANS 1000
ADBIT 16
RGAIN 50
BF 0.05 Hz
T1 0.00
T2 0.00
T3 70.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 490.15 MHz
IRSET 9.16 KHz
IRFIN 7.60 Hz
IRRPW 92 usec
IRATN 79
DFILE AS-2-094-fr3-8-13C.als
SF
LKSET 70.30 KHz
LKFIN 32.5 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.6 c
SLVNT CDCL3
EXREF 77.00 ppm

AS-2-090-fr3-5

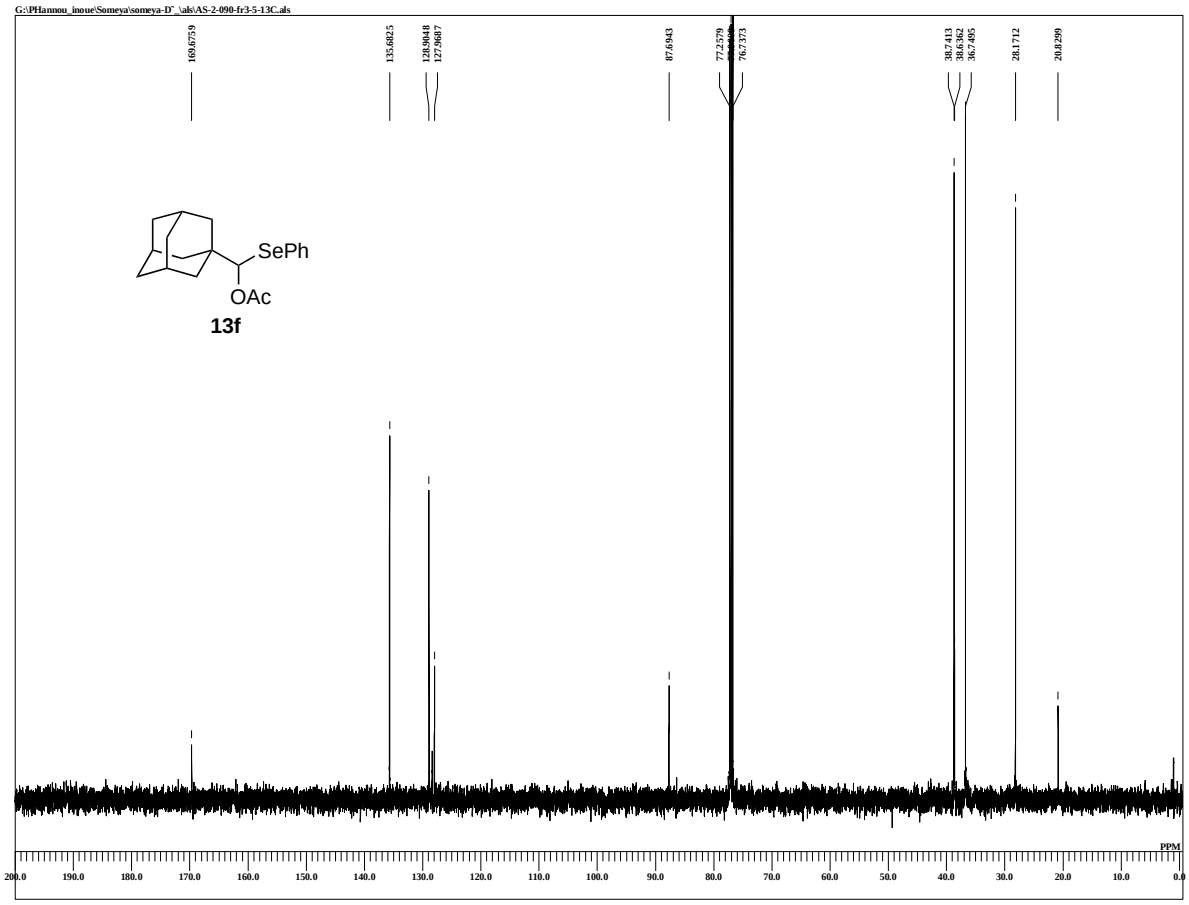
G:\PHannou_inoue\Someya\someya-D^_\als\AS-2-090-fr3-5.als



DFILE AS-2-090-fr3-5.als
COMNT AS-2-090-fr3-5
DATIM 04-04-2009 11:23:32
MENUF
OBNUC 1H
OFR 490.15 MHz
OBFRQ 490.15 MHz
OBSET 9.16 KHz
OBFIN 7.60 Hz
PW1 8.55 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 7352.83 Hz
FLT 37000 Hz
DELAY 13.52 usec
ACQTM 1.7826 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 36
BF 0.01 Hz
T1 0.00
T2 0.00
T3 70.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 490.15 MHz
IRSET 9.16 KHz
IRFIN 7.60 Hz
IRRPW 118 usec
IRATN 79
DFILE AS-2-090-fr3-5.als
SF
LKSET 70.30 KHz
LKFIN 32.5 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 19.5 c
SLVNT CDCL3
EXREF 7.26 ppm

AS-2-090-fr3-5-13C

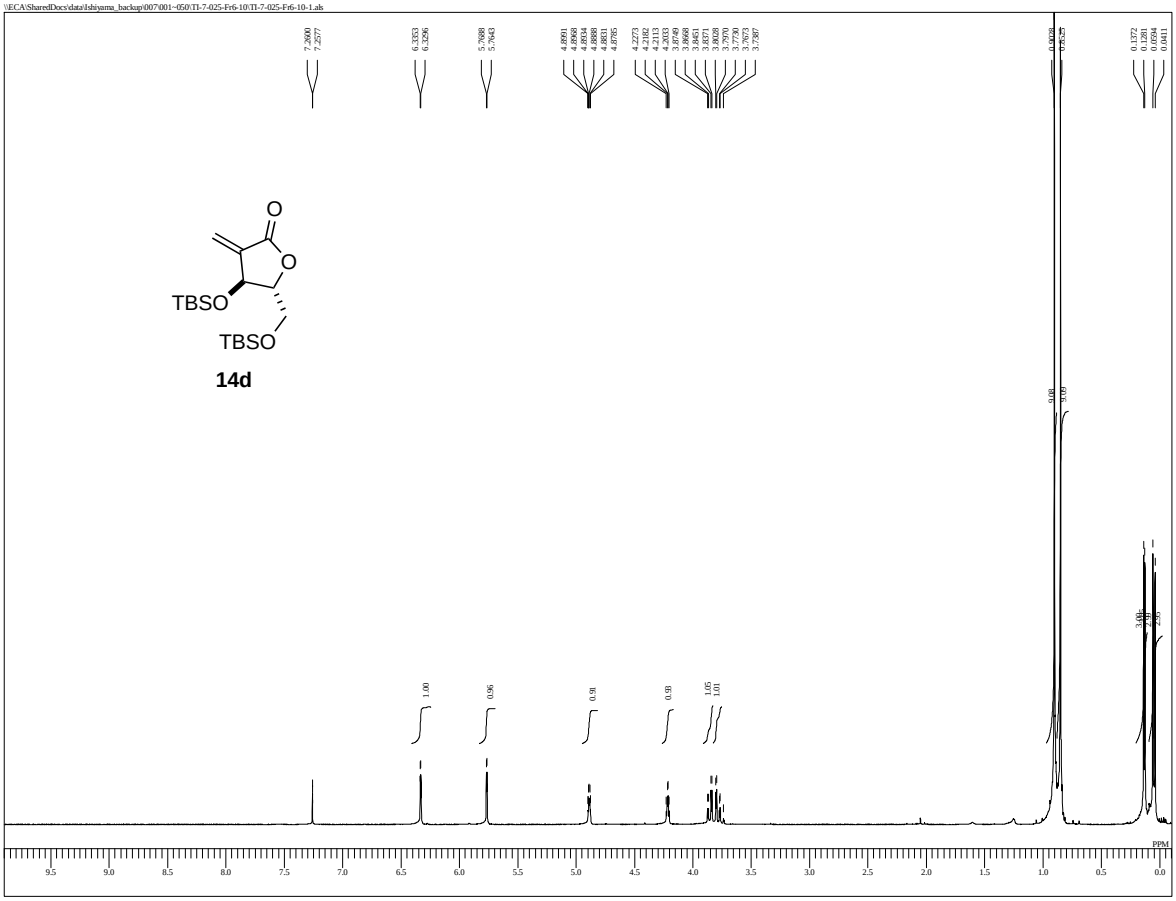
G:\PHannou_inoue\Someya\someya-D^_\als\AS-2-090-fr3-5-13C.als



DFILE AS-2-090-fr3-5-13C.als
COMNT AS-2-090-fr3-5-13C
DATIM 04-04-2009 11:29:33
MENUF
OBNUC 13C
OFR 123.26 MHz
OBFRQ 123.26 MHz
OBSET 2.31 KHz
OBFIN 6.71 Hz
PW1 3.20 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 52428
SPO 52428
TIMES 121
DUMMY 4
FREQU 30863.73 Hz
FLT 155000 Hz
DELAY 21.06 usec
ACQTM 0.8493 sec
PD 2.0000 sec
SCANS 121
ADBIT 16
RGAIN 50
BF 1.00 Hz
T1 0.00
T2 0.00
T3 70.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 490.15 MHz
IRSET 9.16 KHz
IRFIN 7.60 Hz
IRRPW 92 usec
IRATN 79
DFILE AS-2-090-fr3-5-13C.als
SF
LKSET 70.30 KHz
LKFIN 32.5 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.1 c
SLVNT CDCL3
EXREF 77.00 ppm

TI-7-025-Fr6-10

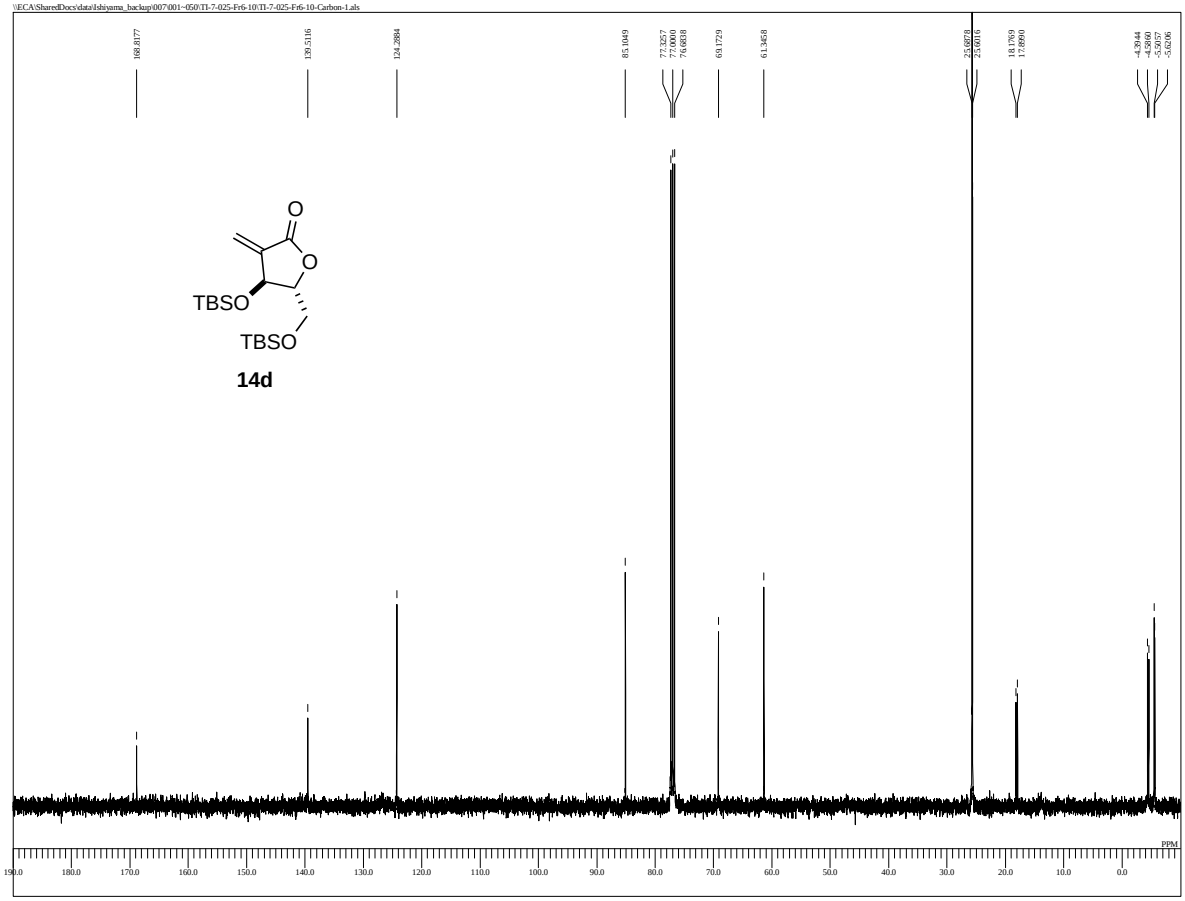
\\ECA\SharedDocs\data\Ishiyama_backup\007\001~050\TI-7-025-Fr6-10\TI-7-025-Fr6-10-1.als



DFILE	TI-7-025-Fr6-10-1.als
COMNT	TI-7-025-Fr6-10
DATIM	09-02-2009 22:16:55
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.50 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	13107
SPO	13107
TIMES	16
DUMMY	1
FREQU	5938.15 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	3.0000 sec
SCANS	16
ADBIT	16
RGAIN	30
BF	0.01 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	TI-7-025-Fr6-10-1.als
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	17.5 c
SLVNT	CDCL3
EXREF	7.26 ppm

TI-7-025-Fr6-10-Carbon

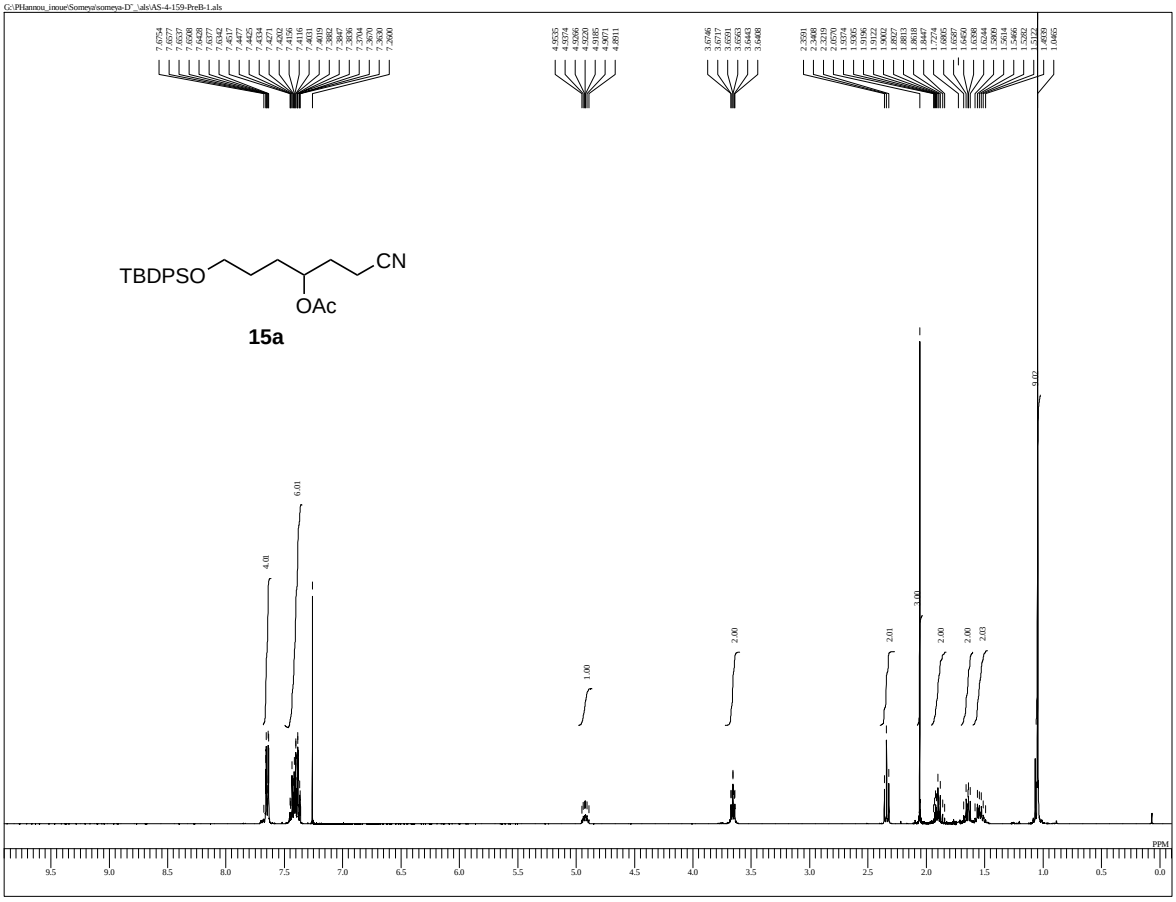
\\ECA\SharedDocs\data\Ishiyama_backup\007\001~050\TI-7-025-Fr6-10\TI-7-025-Fr6-10-Carbon-1.als



DFILE	TI-7-025-Fr6-10-Carbon-1.als
COMNT	TI-7-025-Fr6-10-Carbon
DATIM	09-02-2009 22:28:30
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.25 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	26214
SPO	26214
TIMES	186
DUMMY	4
FREQU	24999.62 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	186
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	TI-7-025-Fr6-10-Carbon-1.als
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	17.7 c
SLVNT	CDCL3
EXREF	77.00 ppm

AS-4-159-PreB

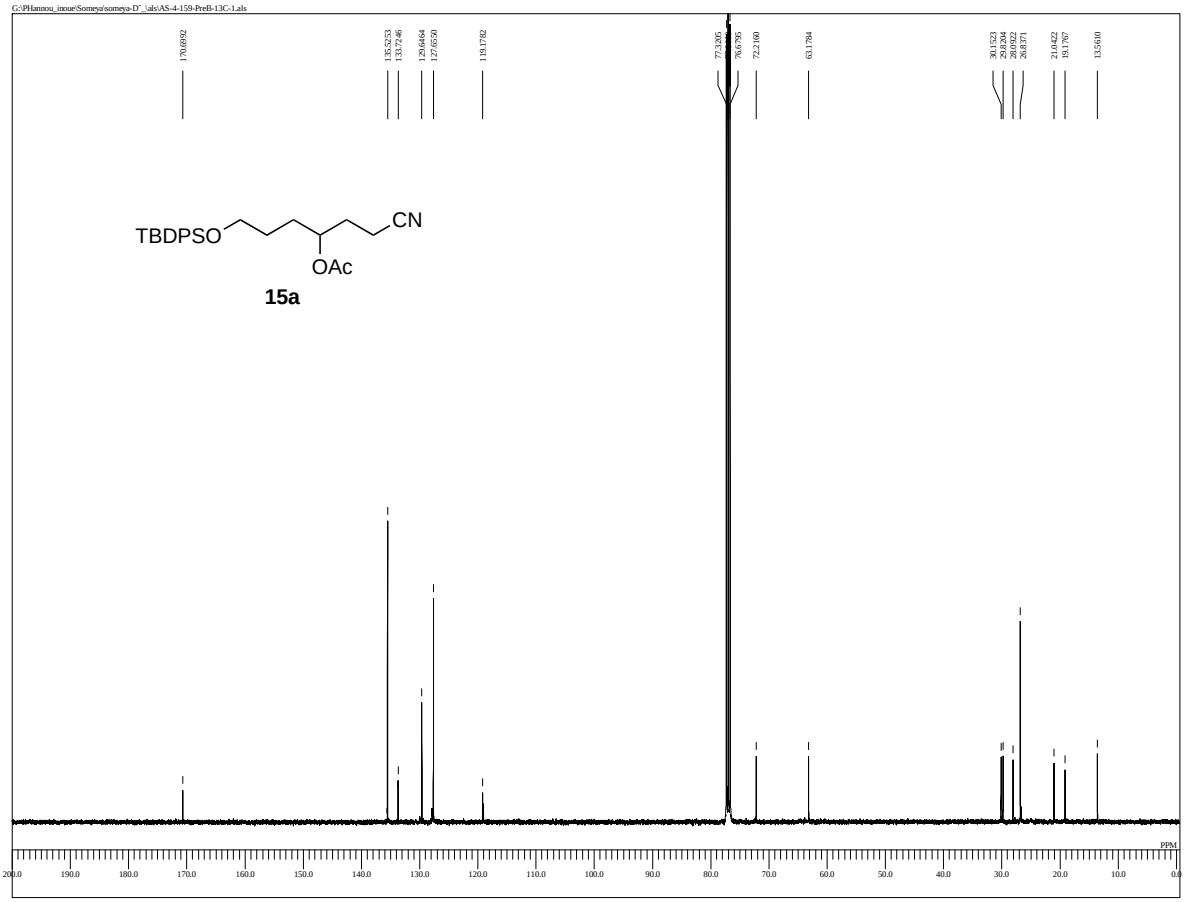
G:\PHannou_inoue\Someya\someya-D'_\als\AS-4-159-PreB-1.als



DFILE AS-4-159-PreB-1.als
COMNT AS-4-159-PreB
DATIM 28-01-2011 22:10:19
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.50 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 26214
SPO 26214
TIMES 16
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 5.0000 sec
SCANS 16
ADBIT 16
RGAIN 40
BF 0.01 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE AS-4-159-PreB-1.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.0 c
SLVNT CDCL3
EXREF 7.26 ppm

AS-4-159-PreB-13C

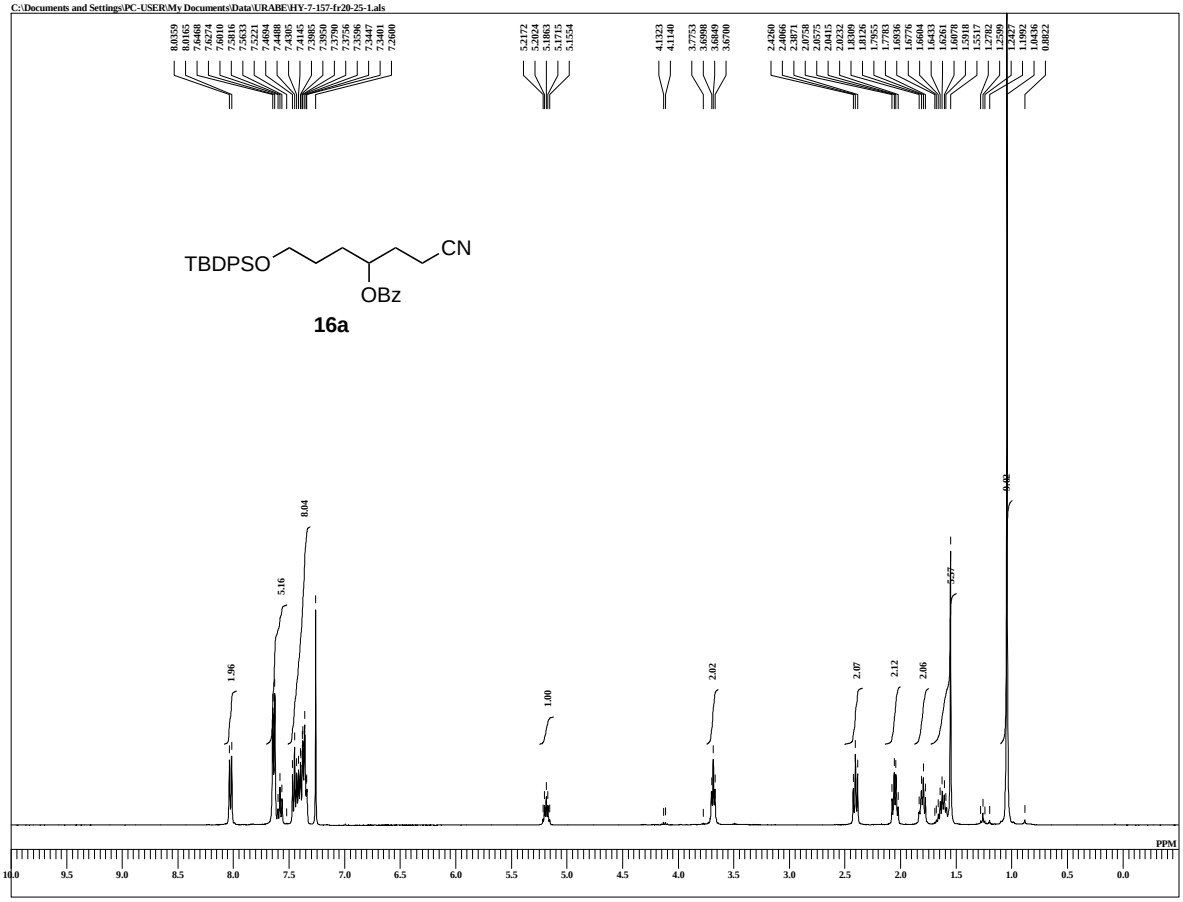
G:\PHannou_inoue\Someya\someya-D'_\als\AS-4-159-PreB-13C-1.als



DFILE AS-4-159-PreB-13C-1.als
COMNT AS-4-159-PreB-13C
DATIM 29-01-2011 00:45:46
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.33 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 65536
SPO 32768
TIMES 2800
DUMMY 4
FREQU 24888.00 Hz
FLT 13000 Hz
DELAY 38.48 usec
ACQTM 1.3166 sec
PD 2.0000 sec
SCANS 2800
ADBIT 16
RGAIN 70
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE AS-4-159-PreB-13C-1.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.0 c
SLVNT CDCL3
EXREF 77.00 ppm

HY-7-157-fr20-25

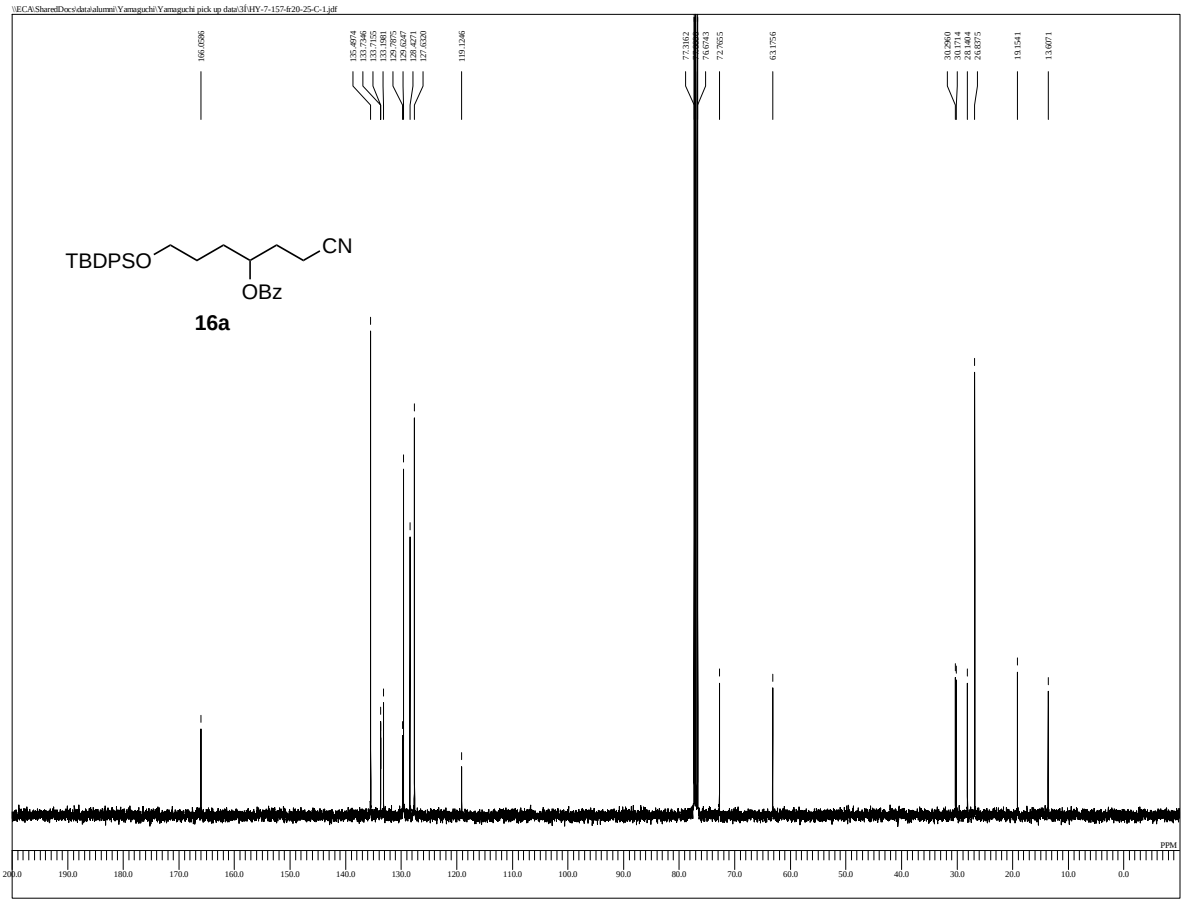
C:\Documents and Settings\PC-USER\My Documents\Data\URABE\HY-7-157-fr20-25-1.als



DFILE HY-7-157-fr20-25-1.als
COMNT HY-7-157-fr20-25
DATIM 29-10-2011 11:51:25
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.62 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 5.0000 sec
SCANS 8
ADBIT 16
RGAIN 46
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE HY-7-157-fr20-25-1.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 25.3 c
SLVNT CDCL3
EXREF 7.26 ppm

HY-7-157-fr20-25-C

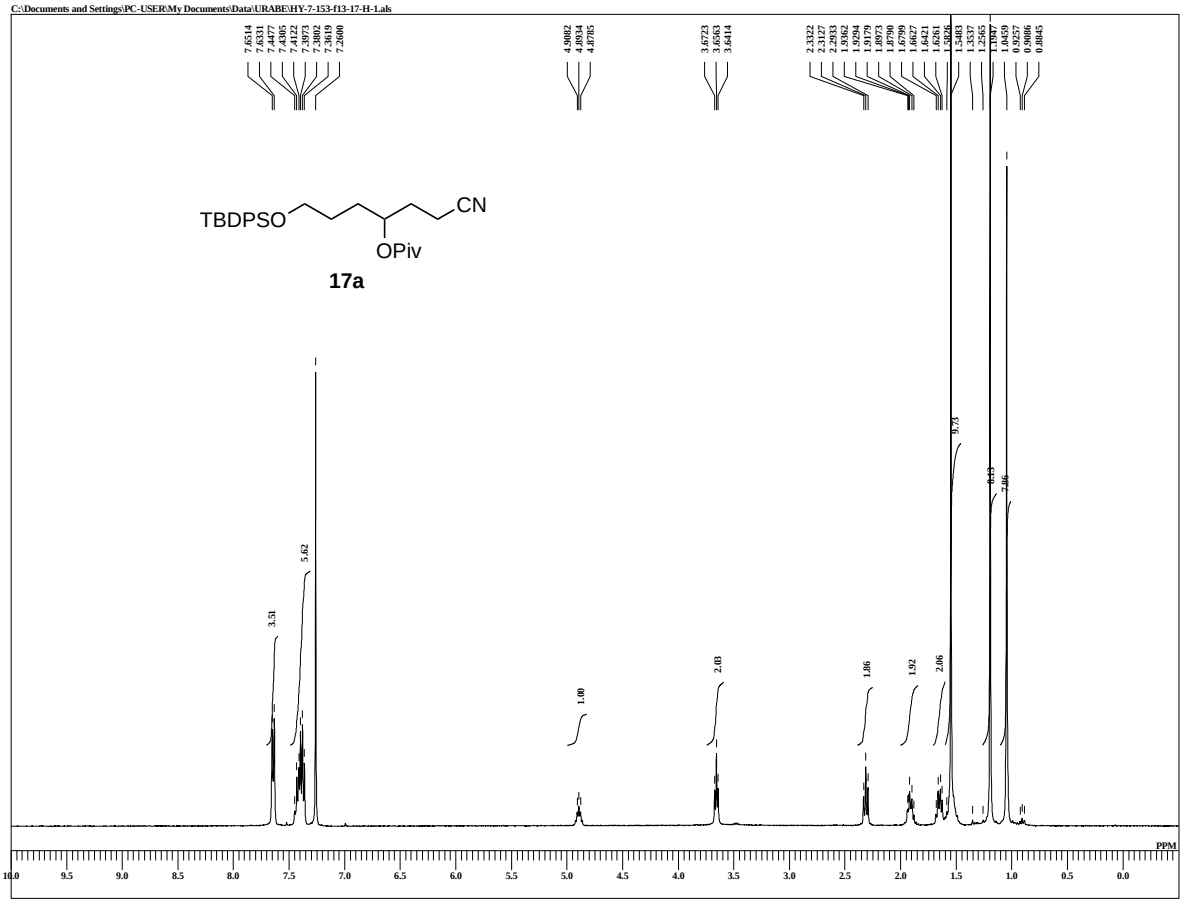
\\ECA\SharedDocs\data\alumni\Yamaguchi\Yamaguchi pick up data\3l\HY-7-157-fr20-25-C-1.jdf



DFILE HY-7-157-fr20-25-C-1.jdf
COMNT HY-7-157-fr20-25-C
DATIM 29-10-2011 12:42:05
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 2.92 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 82
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 8.0000 sec
SCANS 82
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE HY-7-157-fr20-25-C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 25.0 c
SLVNT CDCL3
EXREF 77.00 ppm

HY-7-153-f13-17-H

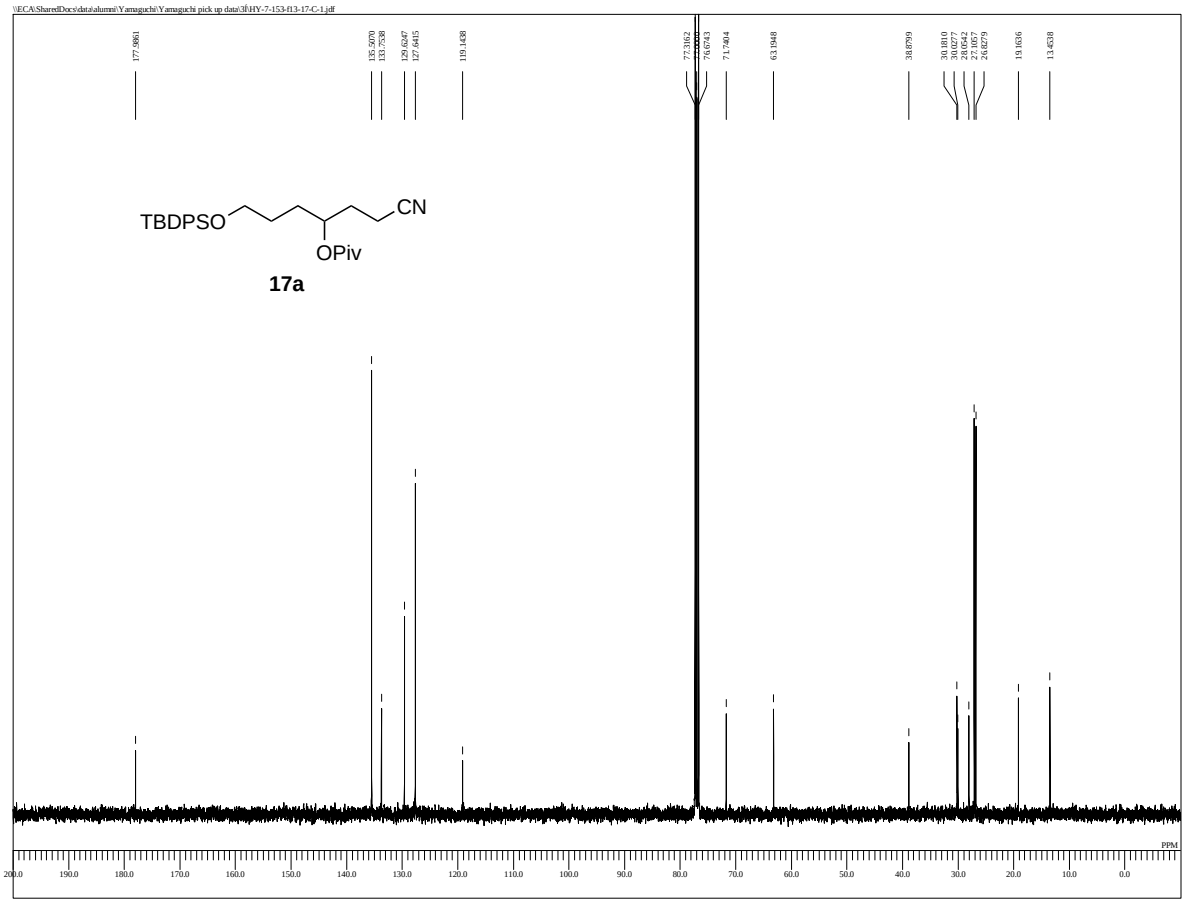
C:\Documents and Settings\PC-USER\My Documents\Data\URABE\HY-7-153-f13-17-H-1.als



DFILE	HY-7-153-f13-17-H-1.als
COMNT	HY-7-153-f13-17-H
DATIM	29-10-2011 17:10:14
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.62 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	13107
SPO	13107
TIMES	8
DUMMY	1
FREQU	5938.15 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	5.0000 sec
SCANS	8
ADBIT	16
RGAIN	50
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	HY-7-153-f13-17-H-1.als
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	25.1 c
SLVNT	CDCL3
EXREF	7.26 ppm

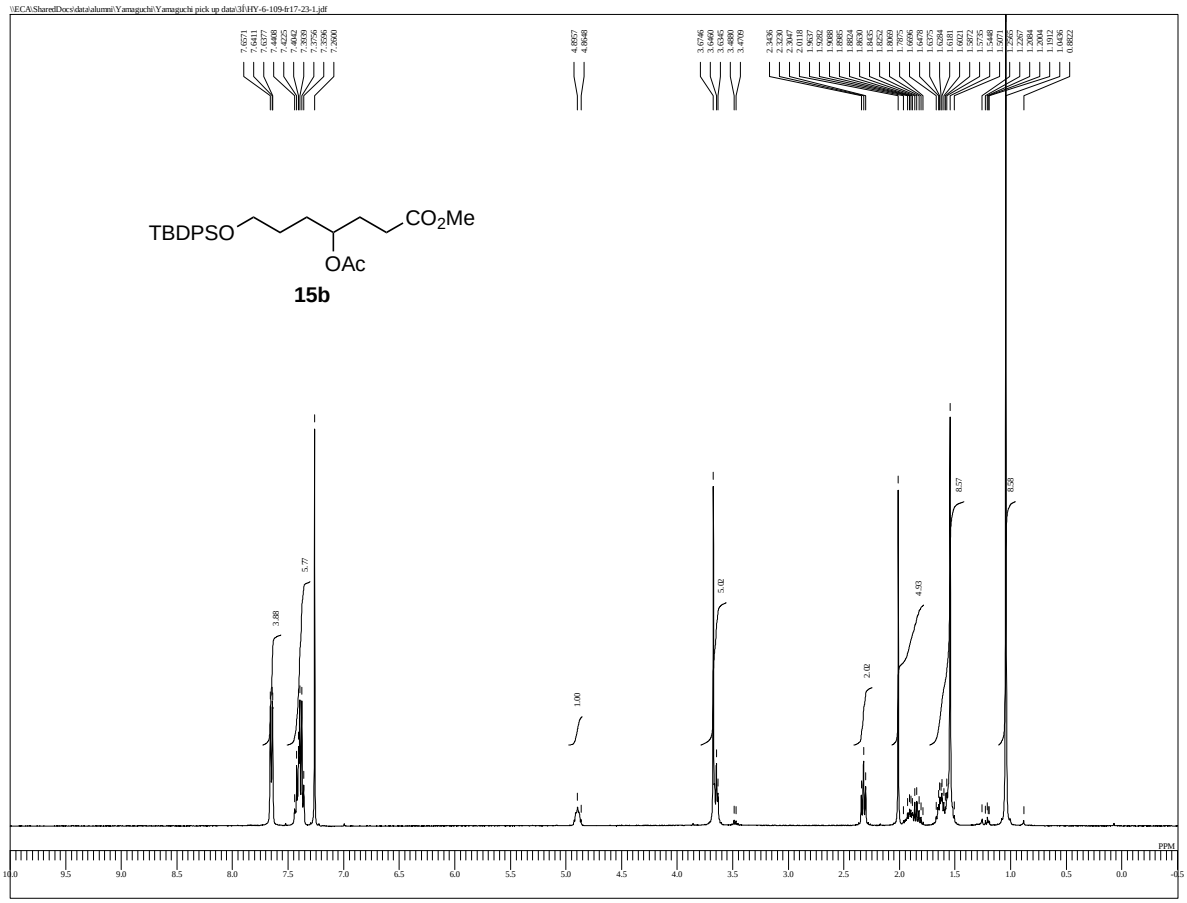
HY-7-153-f13-17-C

\\ECA\SharedDocs\data\alumni\Yamaguchi\Yamaguchi pick up data\3f\HY-7-153-f13-17-C-1.jdf



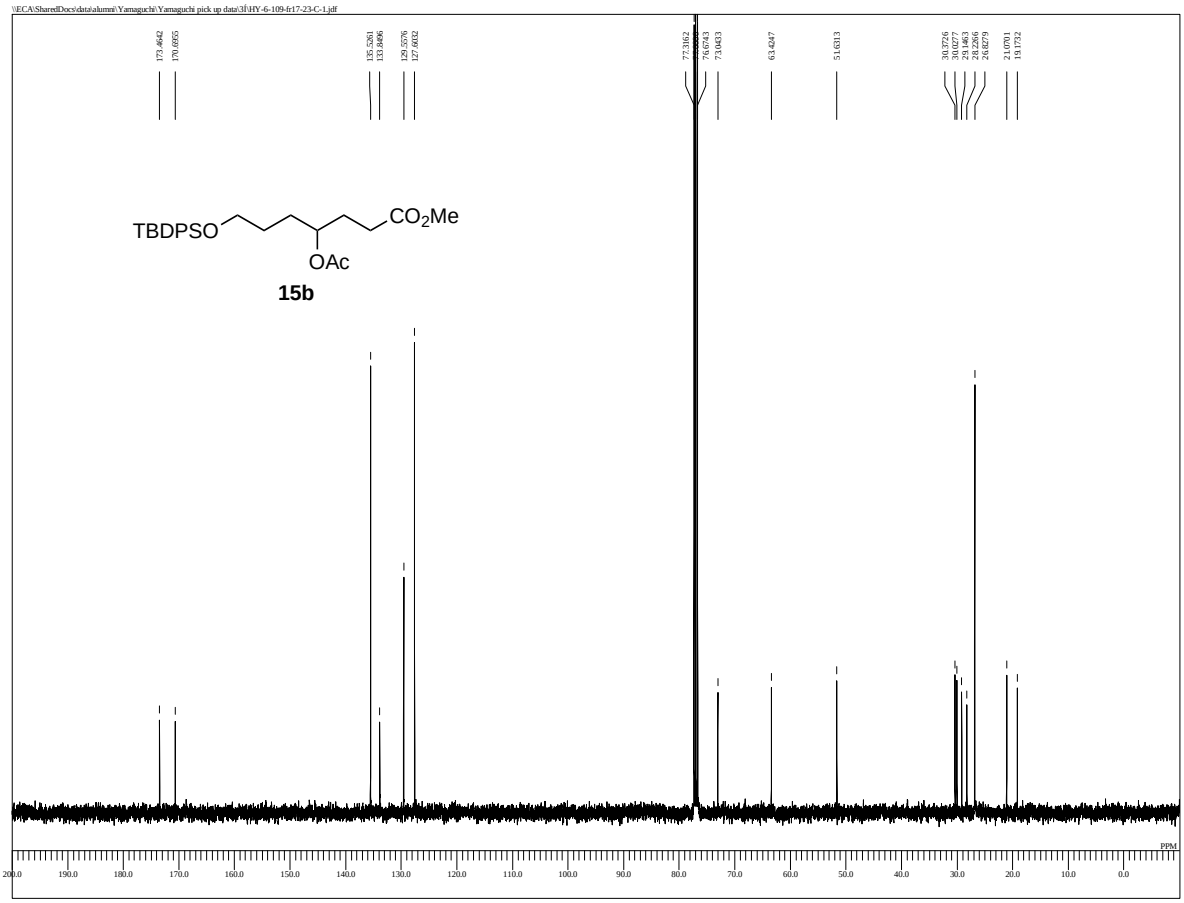
DFILE	HY-7-153-f13-17-C-1.jdf
COMNT	HY-7-153-f13-17-C
DATIM	31-10-2011 10:20:52
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	2.92 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	32768
SPO	32768
TIMES	62
DUMMY	4
FREQU	31250.00 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	8.0000 sec
SCANS	62
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	100.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	HY-7-153-f13-17-C-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	25.2 c
SLVNT	CDCL3
EXREF	77.00 ppm

HY-6-109-fr17-23




```
DFILE     HY-6-109-fr17-23-1.jdf
COMNT     HY-6-109-fr17-23
DATIM     02-11-2011 14:19:53
MENUF
OBNUC     1H
OFR       395.88 MHz
OBFRQ     395.88 MHz
OBSET     6.28 KHz
OBFIN     0.87 Hz
PW1       6.62 usec
DEADT     0.00 usec
PREDL     0.00000 msec
IWT       1.0000 sec
POINT     16384
SPO       16384
TIMES     8
DUMMY     1
FREQU     7422.80 Hz
FLT       30000 Hz
DELAY     16.68 usec
ACQTM     2.2073 sec
PD        5.0000 sec
SCANS     8
ADBIT     16
RGAIN     50
BF        1.00 Hz
T1        0.00
T2        0.00
T3        100.00
T4        100.00
EXMOD     single_pulse.ex2
EXPCM
IRNUC     1H
IFR       395.88 MHz
IRSET     6.28 KHz
IRFIN     0.87 Hz
IRRPW     115 usec
IRATN     79
DFILE     HY-6-109-fr17-23-1.jdf
SF
LKSET     13.20 KHz
LKFIN     75.7 Hz
LKLEV     0
LGAIN     0
LKPHS     0
LKSIG     0
CSPED     0 Hz
FILDC
FILDF
CTEMP     25.2 c
SLVNT     CDCL3
EXREF     7.26 ppm
```

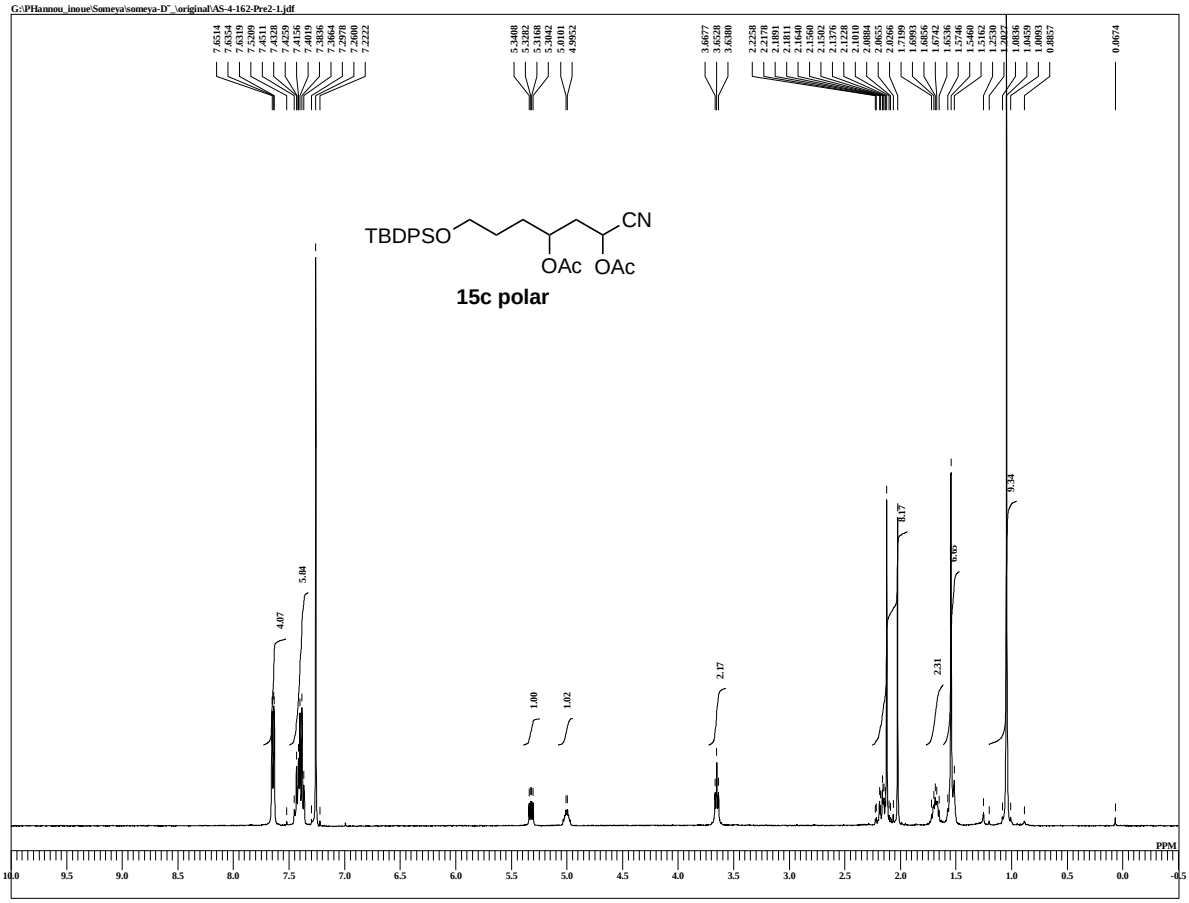
HY-6-109-fr17-23-C




```
DFILE     HY-6-109-fr17-23-C-1.jdf
COMNT     HY-6-109-fr17-23-C
DATIM     02-11-2011 15:55:20
MENUF
OBNUC     13C
OFR       99.55 MHz
OBFRQ     99.55 MHz
OBSET     5.13 KHz
OBFIN     0.98 Hz
PW1       2.92 usec
DEADT     0.00 usec
PREDL     0.00000 msec
IWT       1.0000 sec
POINT     32768
SPO       32768
TIMES     52
DUMMY     4
FREQU     31250.00 Hz
FLT       125000 Hz
DELAY     20.50 usec
ACQTM     1.0486 sec
PD        8.0000 sec
SCANS     52
ADBIT     16
RGAIN     60
BF        1.00 Hz
T1        0.00
T2        0.00
T3        100.00
T4        100.00
EXMOD     single_pulse_dec
EXPCM
IRNUC     1H
IFR       395.88 MHz
IRSET     6.28 KHz
IRFIN     0.87 Hz
IRRPW     115 usec
IRATN     79
DFILE     HY-6-109-fr17-23-C-1.jdf
SF
LKSET     13.20 KHz
LKFIN     75.7 Hz
LKLEV     0
LGAIN     0
LKPHS     0
LKSIG     0
CSPED     0 Hz
FILDC
FILDF
CTEMP     25.3 c
SLVNT     CDCL3
EXREF     77.00 ppm
```

AS-4-162-Pre2

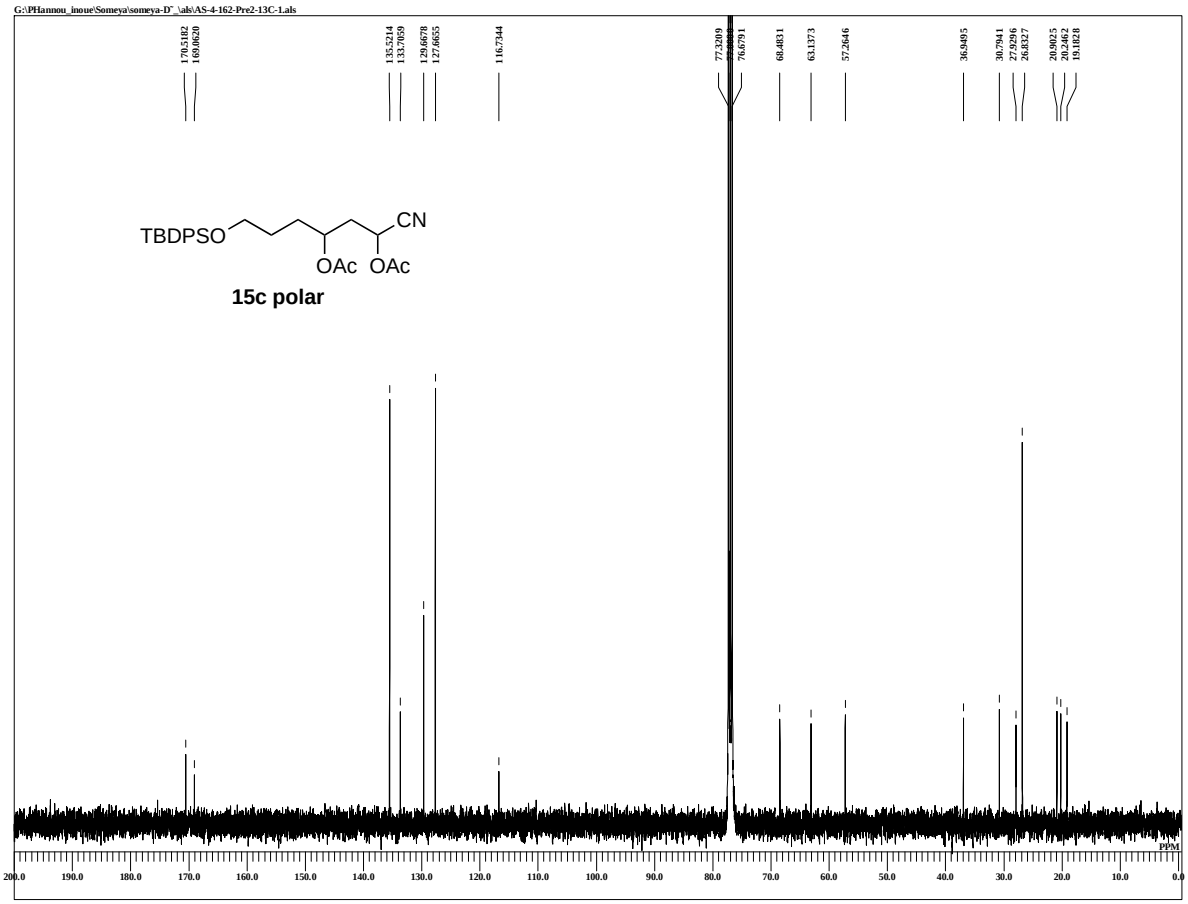
G:\PHannou_inoue\Someya\someya-D^_\original\AS-4-162-Pre2-1.jdf



DFILE	AS-4-162-Pre2-1.jdf
COMNT	AS-4-162-Pre2
DATIM	11-11-2010 16:39:19
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.50 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	54
BF	1.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	AS-4-162-Pre2-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	22.2 c
SLVNT	CDCL3
EXREF	7.26 ppm

AS-4-162-Pre2-13C

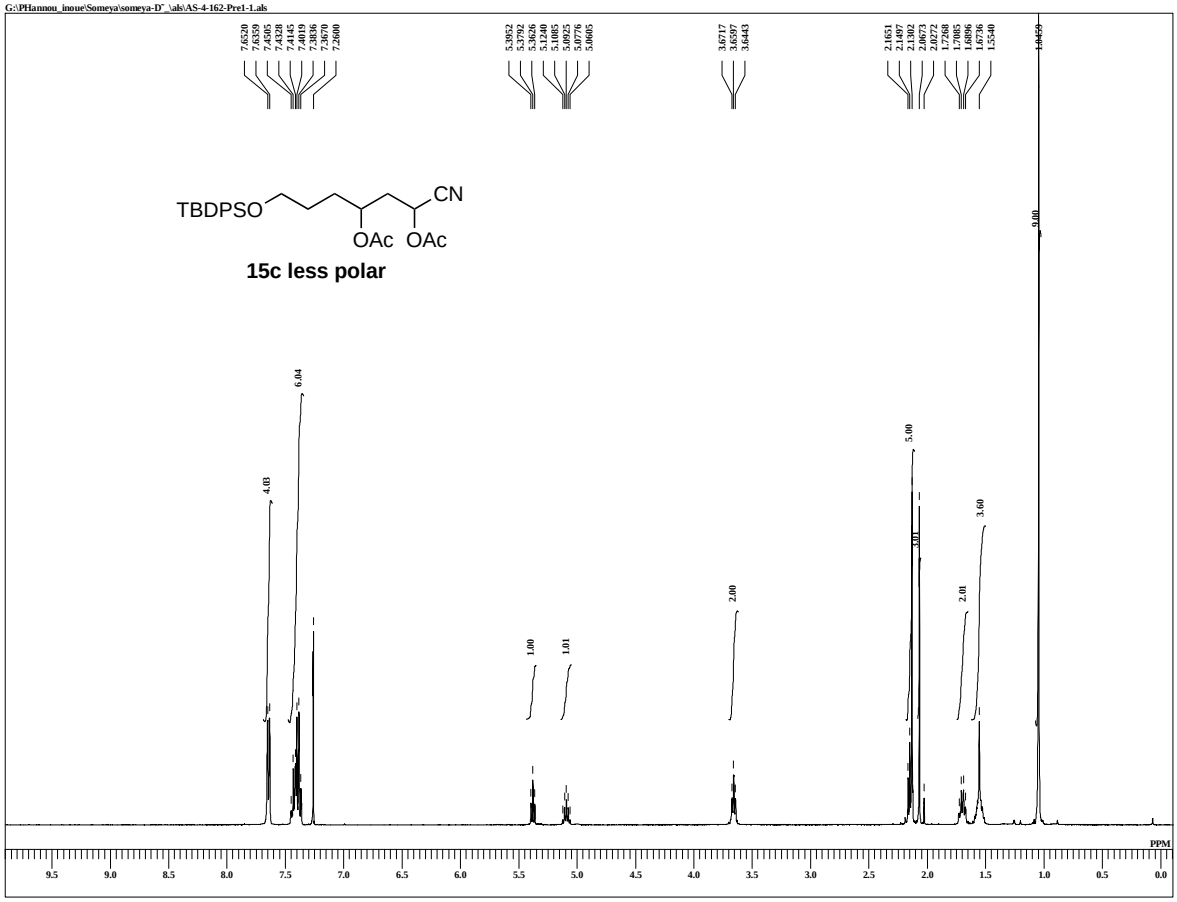
G:\PHannou_inoue\Someya\someya-D^_\als\AS-4-162-Pre2-13C-1.als



DFILE	AS-4-162-Pre2-13C-1.als
COMNT	AS-4-162-Pre2-13C
DATIM	13-11-2010 00:52:55
MENUF	
OBNUC	13C
OFR	99.55 MHz
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
PW1	3.33 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	52428
SPO	52428
TIMES	3400
DUMMY	4
FREQU	24999.62 Hz
FLT	125000 Hz
DELAY	20.50 usec
ACQTM	1.0486 sec
PD	2.0000 sec
SCANS	3400
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	AS-4-162-Pre2-13C-1.als
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	22.4 c
SLVNT	CDCL3
EXREF	77.00 ppm

AS-4-162-Pre1

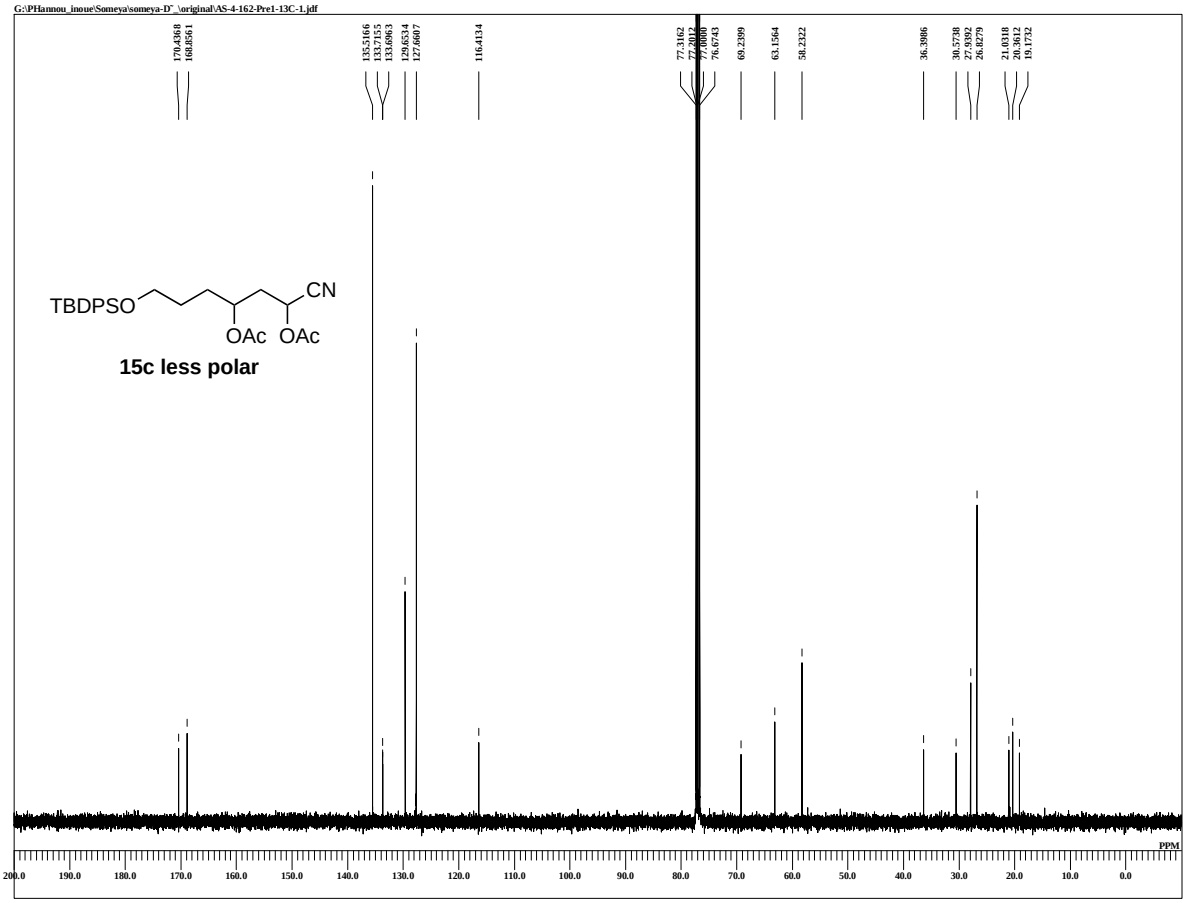
G:\PHannou_inoue\Someya\someya-D^_\als\AS-4-162-Pre1-1.als




```
DFILE    AS-4-162-Pre1-1.als
COMNT    AS-4-162-Pre1
DATIM    11-11-2010 16:36:36
MENUF
OBNUC    1H
OFR      395.88 MHz
OBFRQ    395.88 MHz
OBSET    6.28 KHz
OBFIN    0.87 Hz
PW1      6.50 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    26214
SPO      26214
TIMES    8
DUMMY    1
FREQU    5938.15 Hz
FLT      30000 Hz
DELAY    16.68 usec
ACQTM    2.2073 sec
PD       2.0000 sec
SCANS    8
ADBIT    16
RGAIN    46
BF       0.01 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    AS-4-162-Pre1-1.als
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    22.2 c
SLVNT    CDCL3
EXREF    7.26 ppm
```

AS-4-162-Pre1-13C

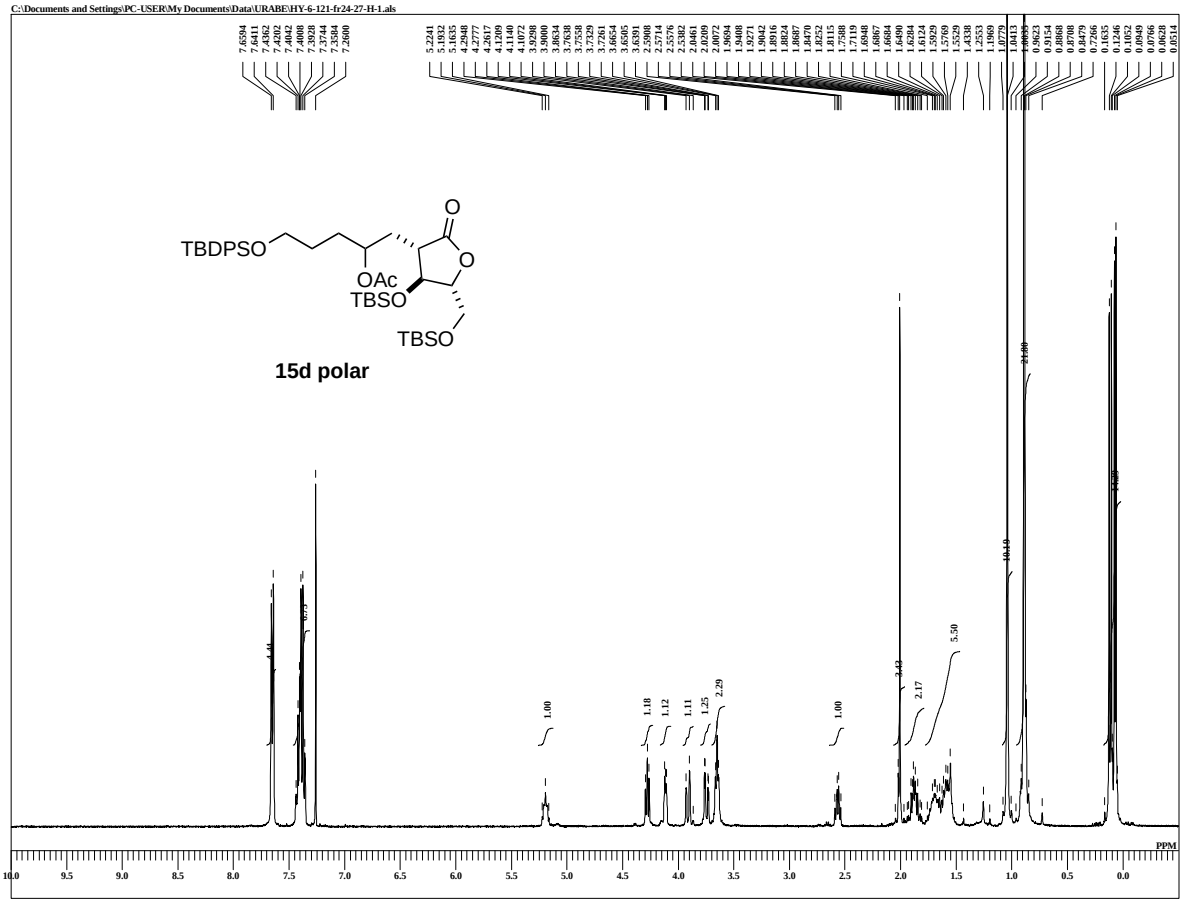
G:\PHannou_inoue\Someya\someya-D^_\original\AS-4-162-Pre1-13C-1.jdf




```
DFILE    AS-4-162-Pre1-13C-1.jdf
COMNT    AS-4-162-Pre1-13C
DATIM    16-11-2010 00:39:34
MENUF
OBNUC    13C
OFR      99.55 MHz
OBFRQ    99.55 MHz
OBSET    5.13 KHz
OBFIN    0.98 Hz
PW1      3.33 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    32768
SPO      32768
TIMES    3400
DUMMY    4
FREQU    31250.00 Hz
FLT      125000 Hz
DELAY    20.50 usec
ACQTM    1.0486 sec
PD       2.0000 sec
SCANS    3400
ADBIT    16
RGAIN    60
BF       0.01 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    AS-4-162-Pre1-13C-1.jdf
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    22.5 c
SLVNT    CDCL3
EXREF    77.00 ppm
```

HY-6-121-fr24-27-H

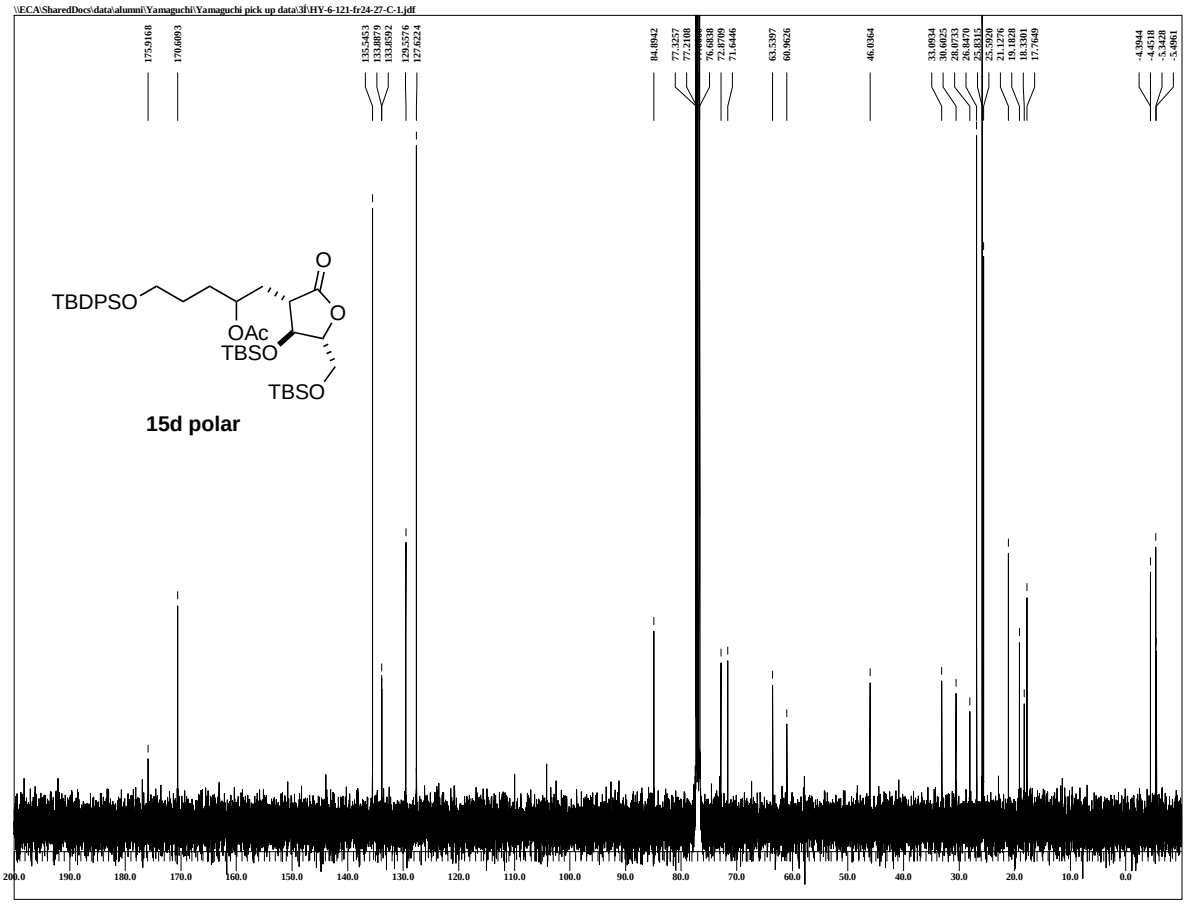
C:\Documents and Settings\PC-USER\My Documents\Data\URABE\HY-6-121-fr24-27-H-1.als



DFILE HY-6-121-fr24-27-H-1.als
COMNT HY-6-121-fr24-27-H
DATIM 29-01-2012 21:40:47
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.62 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 5.0000 sec
SCANS 8
ADBIT 16
RGAIN 38
BF 0.01 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE HY-6-121-fr24-27-H-1.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 22.5 c
SLVNT CDCL3
EXREF 7.26 ppm

HY-6-121-fr24-27-C

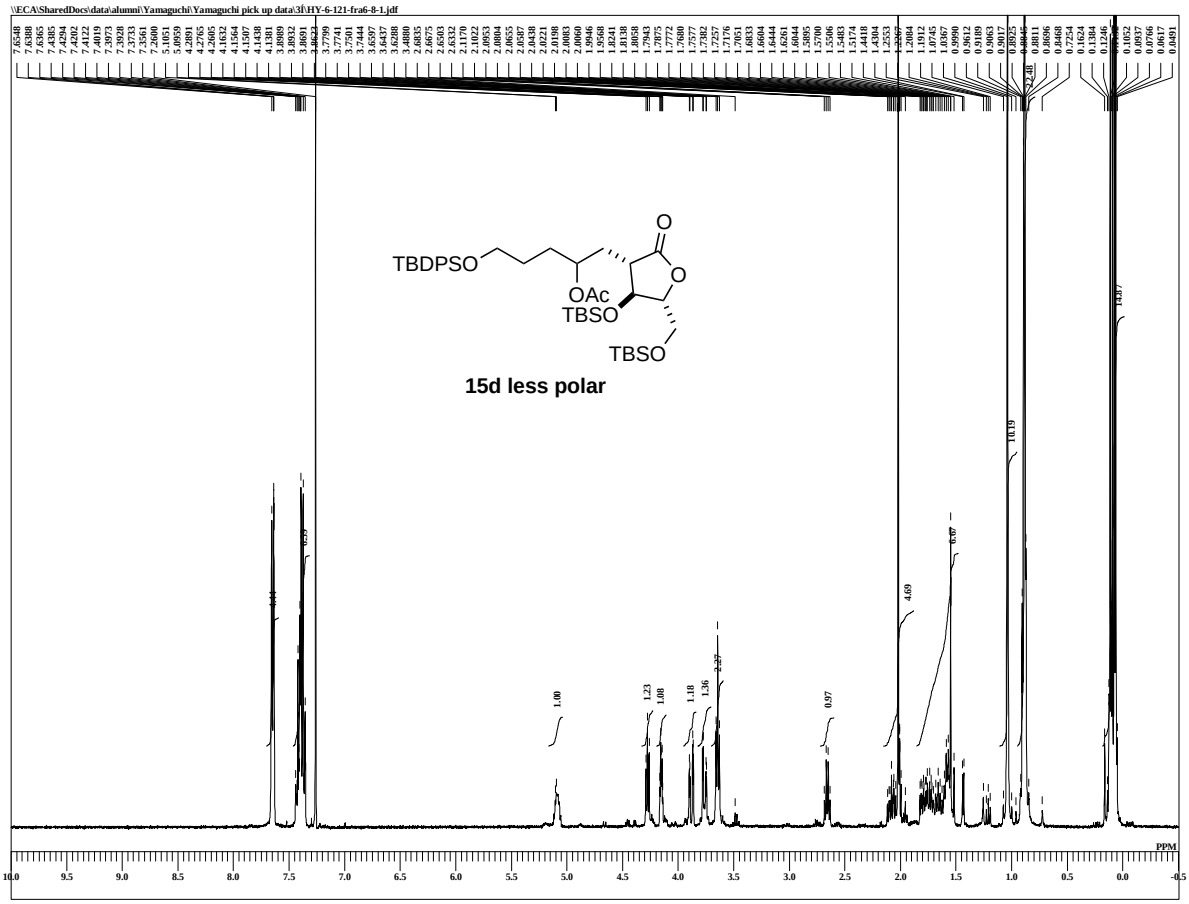
\\ECA\SharedDocs\data\alumni\Yamaguchi\Yamaguchi pick up data\3f\HY-6-121-fr24-27-C-1.jdf



DFILE HY-6-121-fr24-27-C-1.jdf
COMNT HY-6-121-fr24-27-C
DATIM 30-01-2012 03:47:03
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 2.92 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 2400
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 8.0000 sec
SCANS 2400
ADBIT 16
RGAIN 60
BF 0.01 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE HY-6-121-fr24-27-C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.1 c
SLVNT CDCL3
EXREF 77.00 ppm

HY-6-121-fra6-8

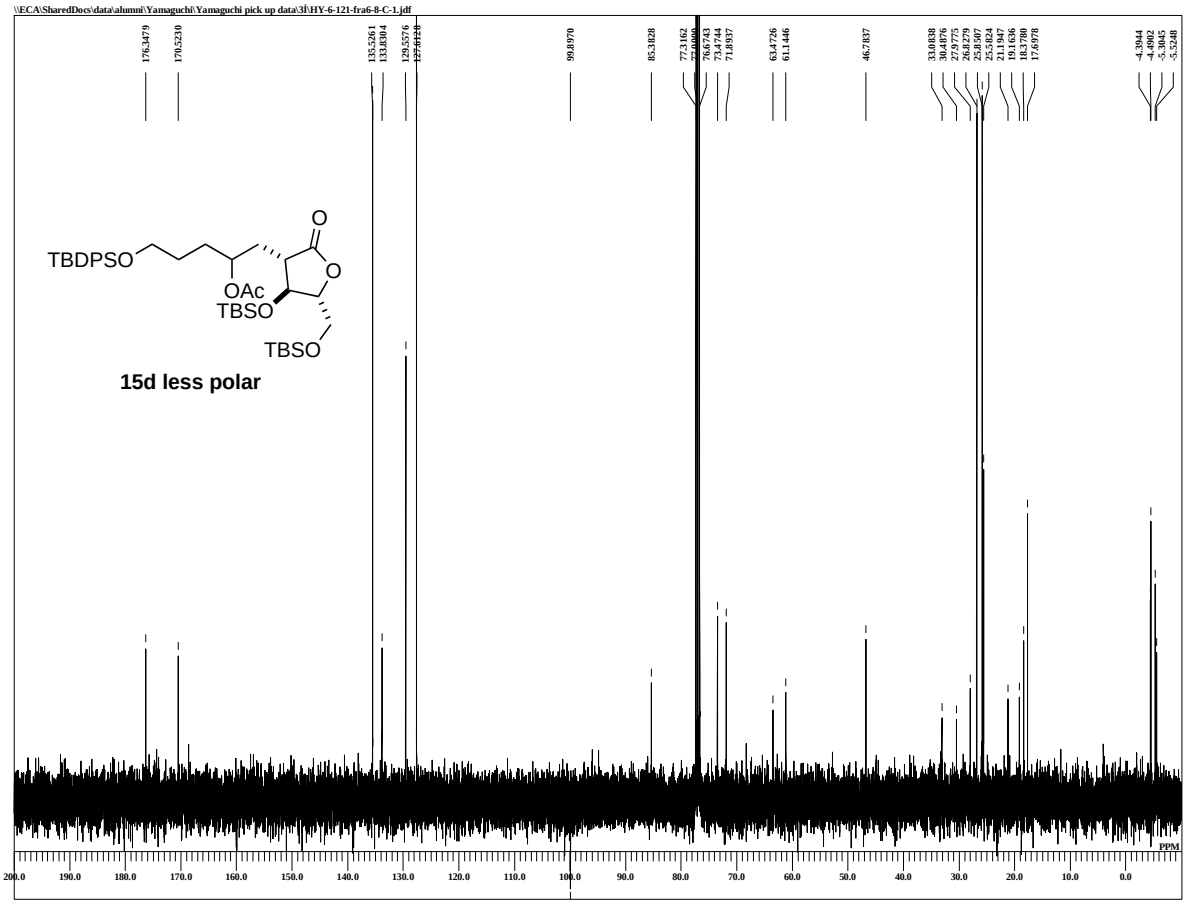
\\ECA\SharedDocs\data\alumni\Yamaguchi\Yamaguchi pick up data\3f\HY-6-121-fra6-8-1.jdf




```
DFILE    HY-6-121-fra6-8-1.jdf
COMNT    HY-6-121-fra6-8
DATIM    04-02-2012 11:39:20
MENUF
OBNUC    1H
OFR      395.88 MHz
OBFRQ    395.88 MHz
OBSET    6.28 KHz
OBFIN    0.87 Hz
PW1      6.62 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    16384
SPO      16384
TIMES    8
DUMMY    1
FREQU    7422.80 Hz
FLT      30000 Hz
DELAY    16.68 usec
ACQTM    2.2073 sec
PD       5.0000 sec
SCANS    8
ADBIT    16
RGAIN    42
BF       0.01 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    HY-6-121-fra6-8-1.jdf
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    22.8 c
SLVNT    CDCL3
EXREF    7.26 ppm
```

HY-6-121-fra6-8-C

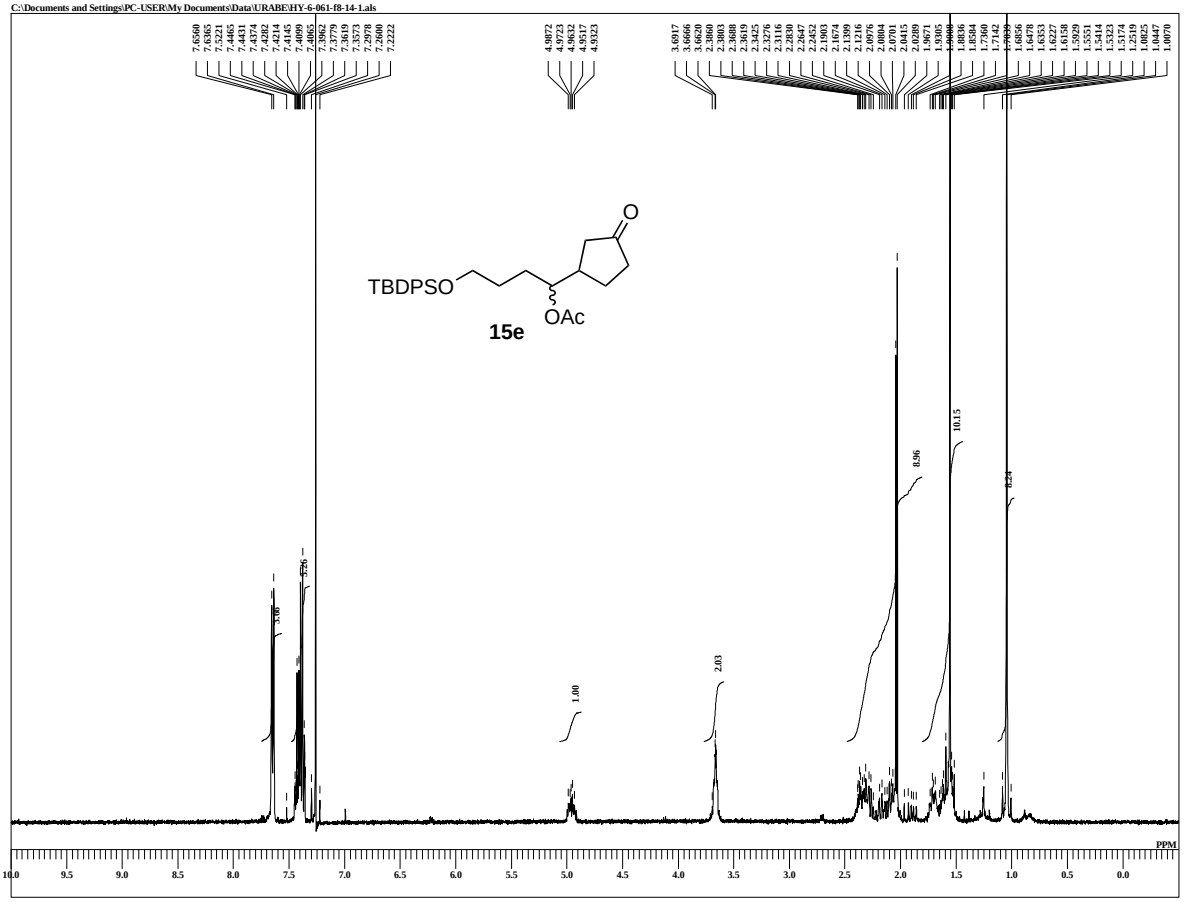
\\ECA\SharedDocs\data\alumni\Yamaguchi\Yamaguchi pick up data\3f\HY-6-121-fra6-8-C-1.jdf




```
DFILE    HY-6-121-fra6-8-C-1.jdf
COMNT    HY-6-121-fra6-8-C
DATIM    06-02-2012 23:25:31
MENUF
OBNUC    13C
OFR      99.55 MHz
OBFRQ    99.55 MHz
OBSET    5.13 KHz
OBFIN    0.98 Hz
PW1      2.92 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    32768
SPO      32768
TIMES    226
DUMMY    4
FREQU    31250.00 Hz
FLT      125000 Hz
DELAY    20.50 usec
ACQTM    1.0486 sec
PD       8.0000 sec
SCANS    226
ADBIT    16
RGAIN    60
BF       0.01 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    HY-6-121-fra6-8-C-1.jdf
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    22.8 c
SLVNT    CDCL3
EXREF    77.00 ppm
```

HY-6-061-f8-14

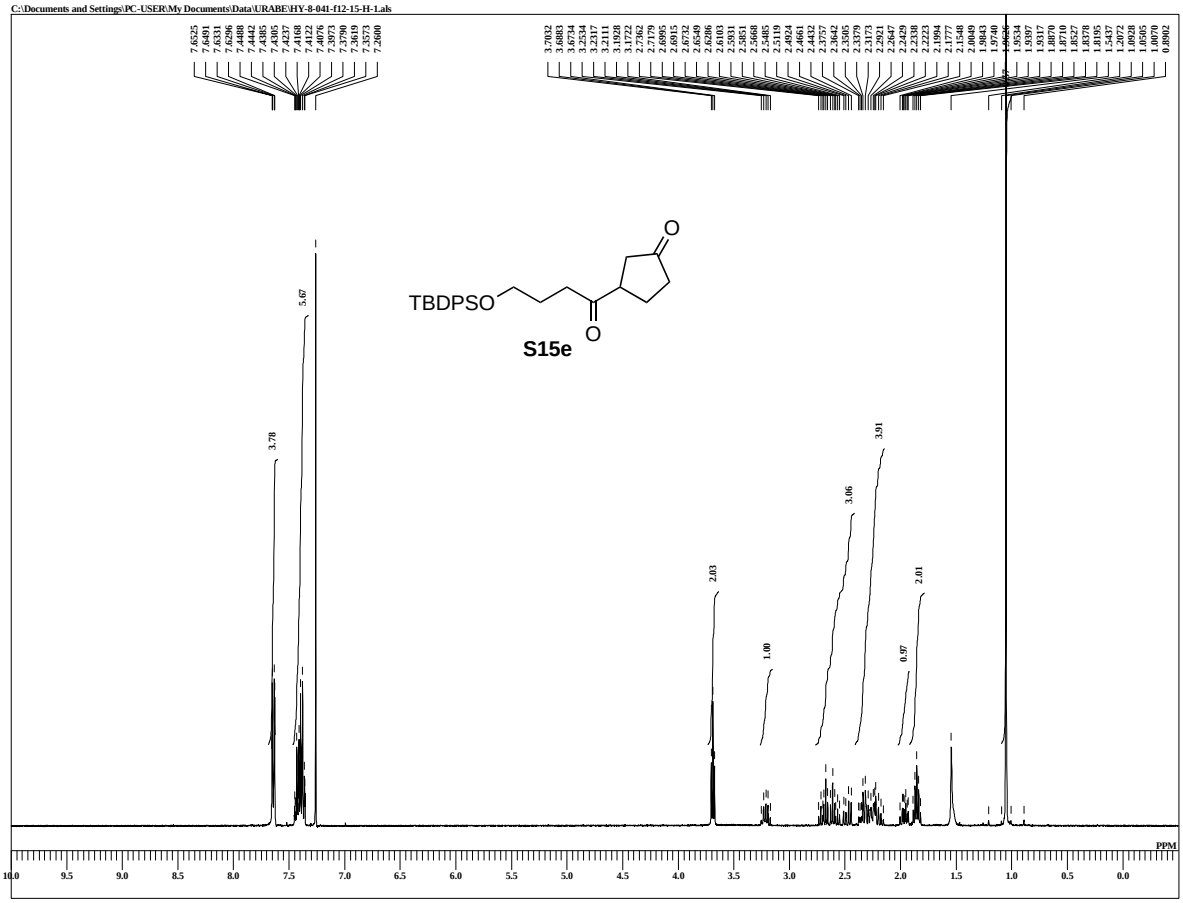
C:\Documents and Settings\PC-USER\My Documents\Data\URABE\HY-6-061-f8-14-1.als



DFILE	HY-6-061-f8-14-1.als
COMNT	HY-6-061-f8-14
DATIM	22-04-2011 10:15:07
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.50 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	13107
SPO	13107
TIMES	8
DUMMY	1
FREQU	5938.15 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	50
BF	0.01 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	HY-6-061-f8-14-1.als
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.0 c
SLVNT	CDCL3
EXREF	7.26 ppm

HY-8-041-f12-15-H

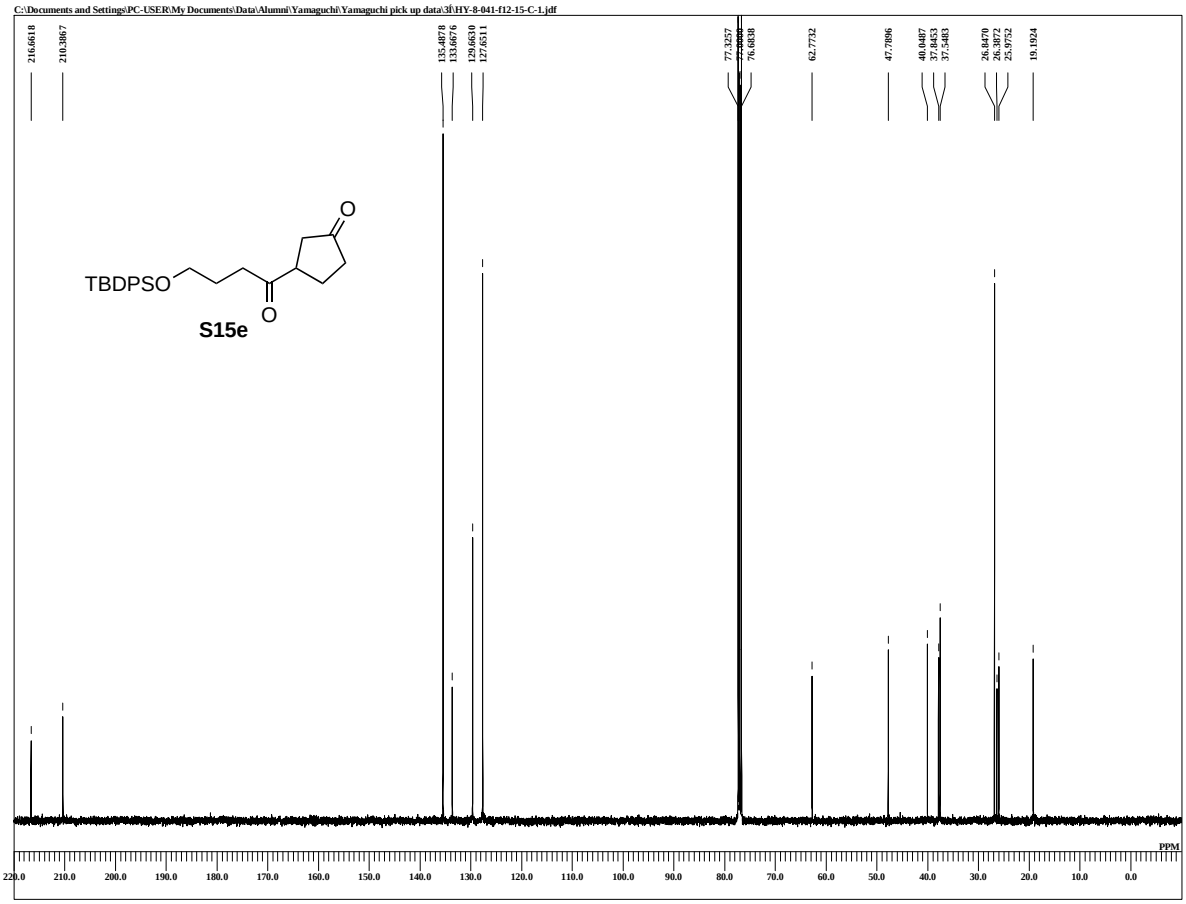
C:\Documents and Settings\PC-USER\My Documents\Data\URABE\HY-8-041-f12-15-H-1.als



DFILE HY-8-041-f12-15-H-1.als
COMNT HY-8-041-f12-15-H
DATIM 09-11-2011 12:40:58
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.62 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 5.0000 sec
SCANS 8
ADBIT 16
RGAIN 50
BF 0.01 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE HY-8-041-f12-15-H-1.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 25.0 c
SLVNT CDCL3
EXREF 7.26 ppm

HY-8-041-f12-15-C

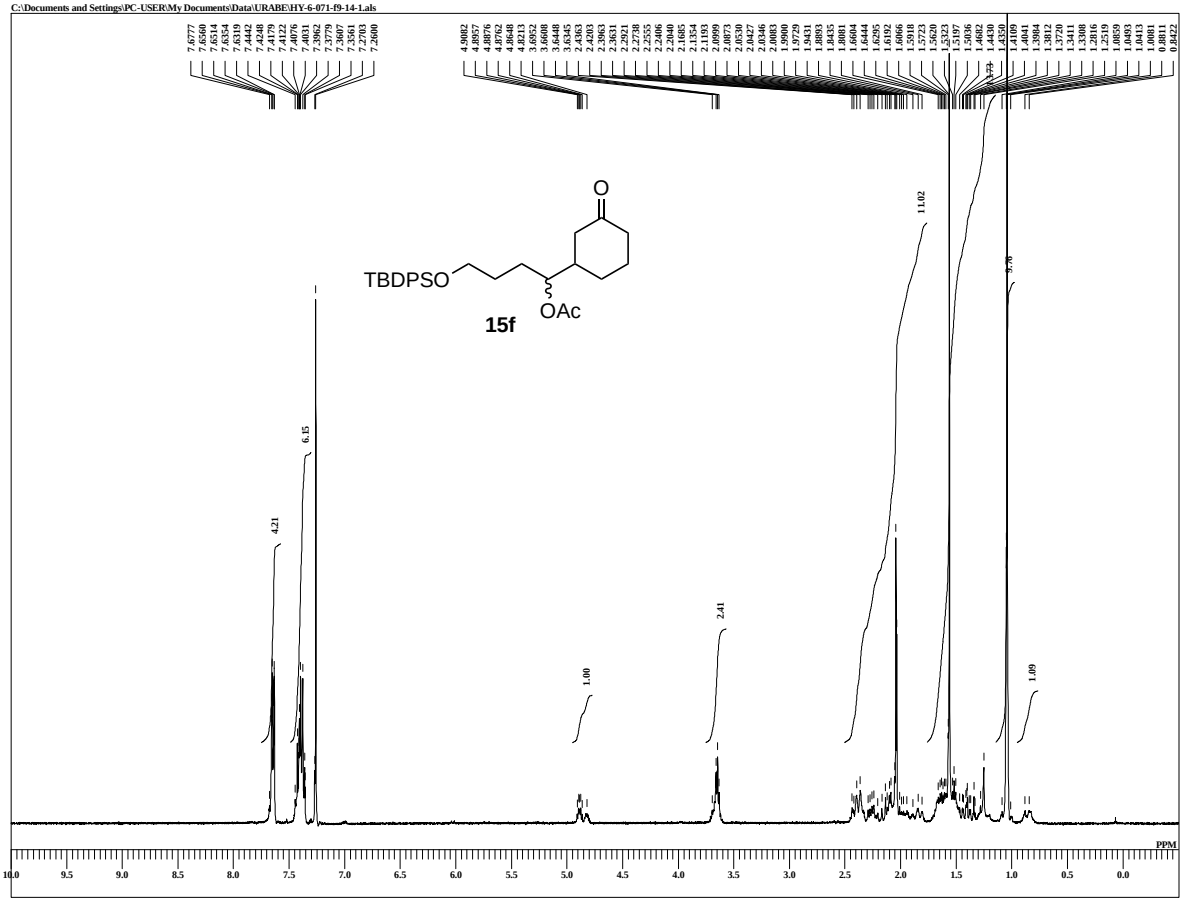
C:\Documents and Settings\PC-USER\My Documents\Data\Alumni\Yamaguchi\Yamaguchi pick up data\3f\HY-8-041-f12-15-C-1.jdf



DFILE HY-8-041-f12-15-C-1.jdf
COMNT HY-8-041-f12-15-C
DATIM 10-11-2011 23:48:42
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 2.92 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 200
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 8.0000 sec
SCANS 200
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE HY-8-041-f12-15-C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 24.7 c
SLVNT CDCL3
EXREF 77.00 ppm

HY-071-f9-14

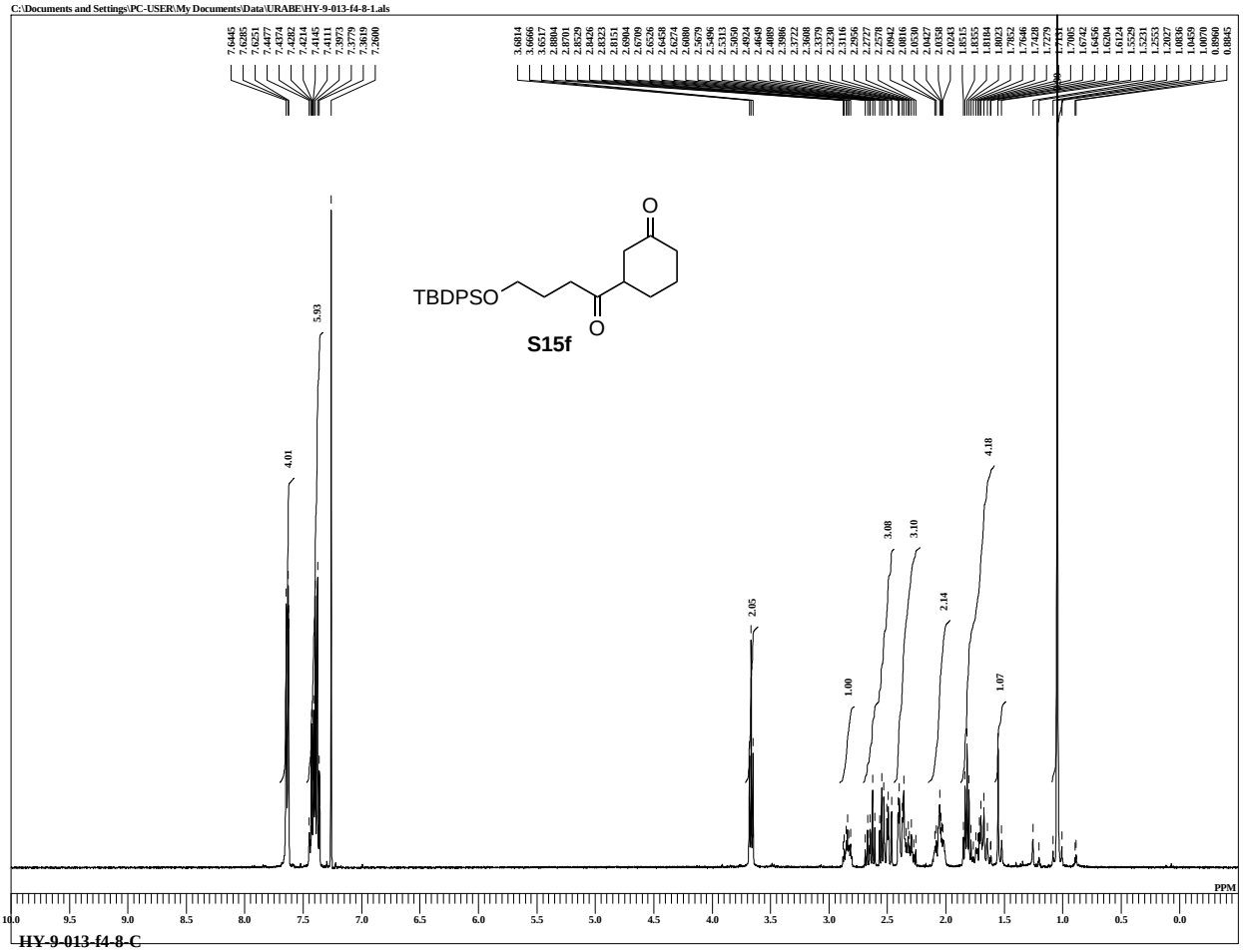
C:\Documents and Settings\PC-USER\My Documents\Data\URABE\HY-6-071-f9-14-1.als



DFILE	HY-6-071-f9-14-1.als
COMNT	HY-071-f9-14
DATIM	27-04-2011 13:57:18
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.50 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	13107
SPO	13107
TIMES	8
DUMMY	1
FREQU	5938.15 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	50
BF	0.01 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	HY-6-071-f9-14-1.als
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	7.26 ppm

HY-9-013-f4-8

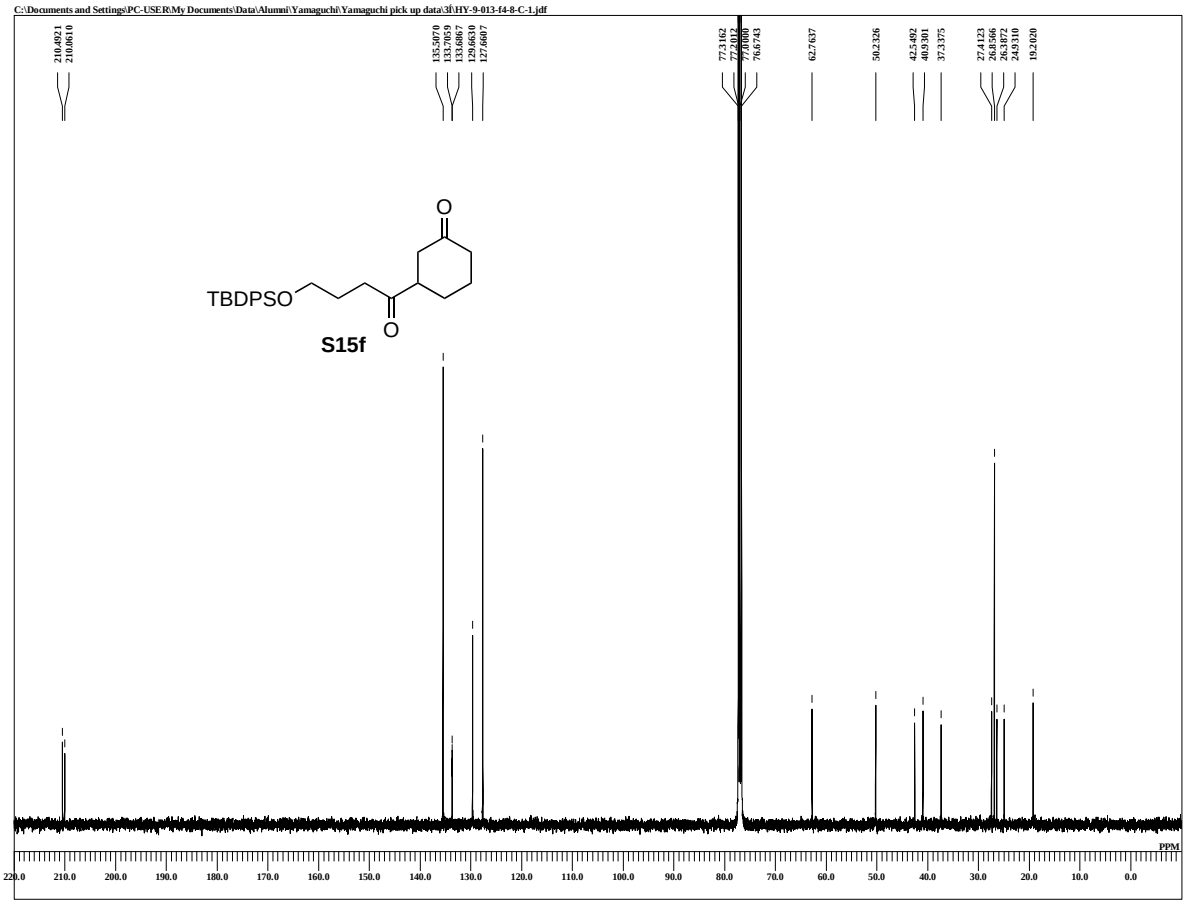
C:\Documents and Settings\PC-USER\My Documents\Data\URABE\HY-9-013-f4-8-1.als



DFILE HY-9-013-f4-8-1.als
COMNT HY-9-013-f4-8
DATIM 10-02-2012 14:25:28
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.62 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 5.0000 sec
SCANS 8
ADBIT 16
RGAIN 46
BF 0.01 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE HY-9-013-f4-8-1.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.0 c
SLVNT CDCL3
EXREF 7.26 ppm

HY-9-013-f4-8-C

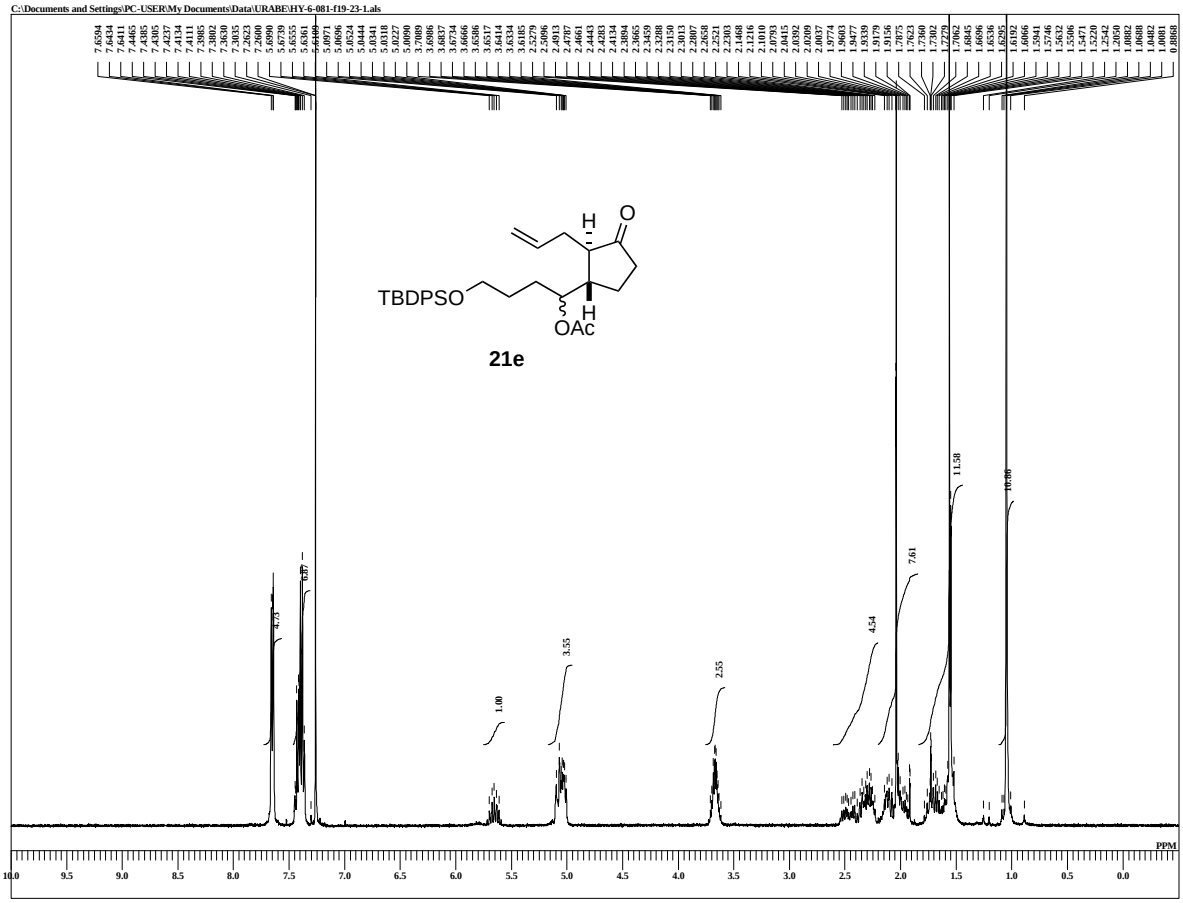
C:\Documents and Settings\PC-USER\My Documents\Data\Alumni\Yamaguchi\Yamaguchi pick up data\3f\HY-9-013-f4-8-C-1.jdf



DFILE HY-9-013-f4-8-C-1.jdf
COMNT HY-9-013-f4-8-C
DATIM 13-02-2012 02:50:00
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 2.92 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 2000
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 8.0000 sec
SCANS 2000
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE HY-9-013-f4-8-C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 22.9 c
SLVNT CDCL3
EXREF 77.00 ppm

HY-6-081-f19-23

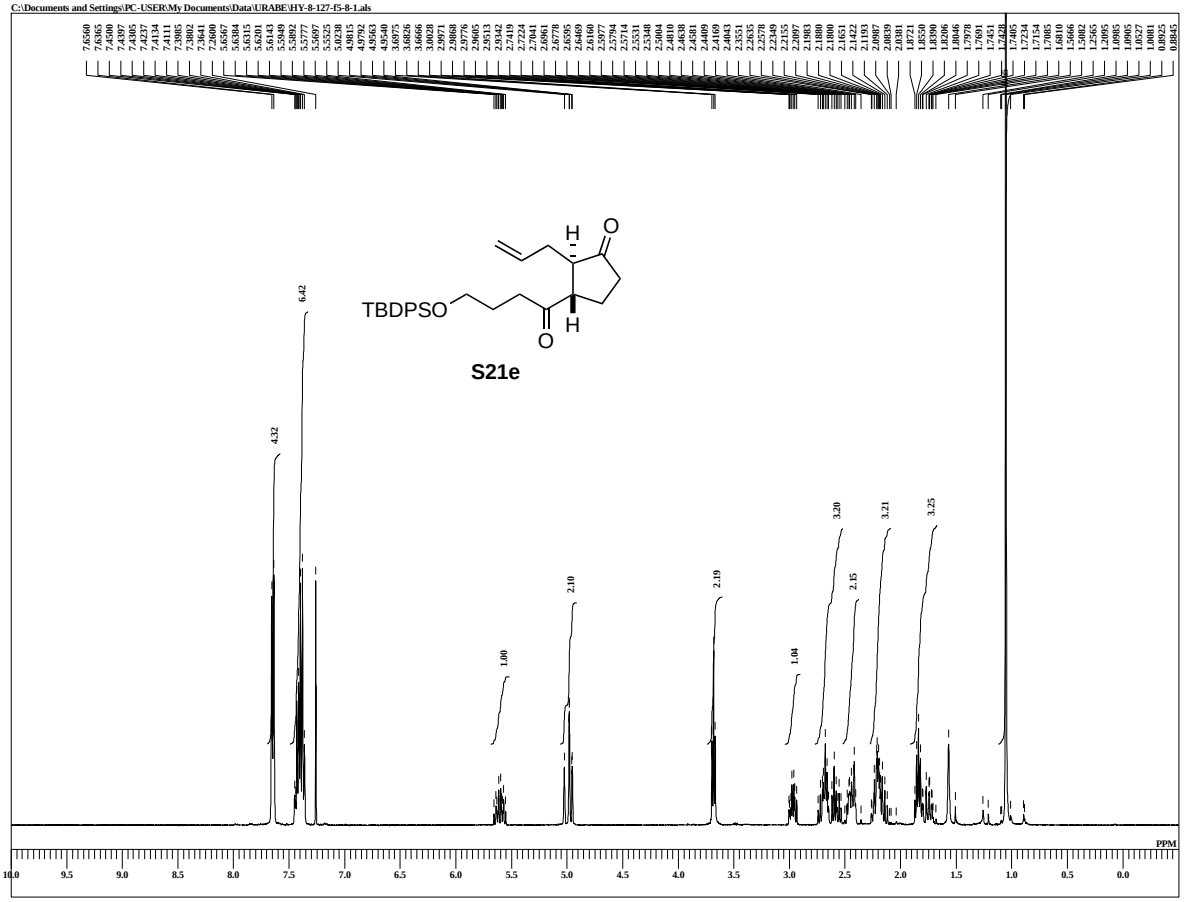
C:\Documents and Settings\PC-USER\My Documents\Data\URABE\HY-6-081-f19-23-1.als



DFILE	HY-6-081-f19-23-1.als
COMNT	HY-6-081-f19-23
DATIM	02-05-2011 10:33:22
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.50 usec
DEADT	0.00 usec
PREDL	0.00000 usec
IWT	1.0000 sec
POINT	13107
SPO	13107
TIMES	8
DUMMY	1
FREQU	5938.15 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	50
BF	0.01 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	HY-6-081-f19-23-1.als
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	20.0 c
SLVNT	CDCL3
EXREF	7.26 ppm

HY-8-127-f5-8

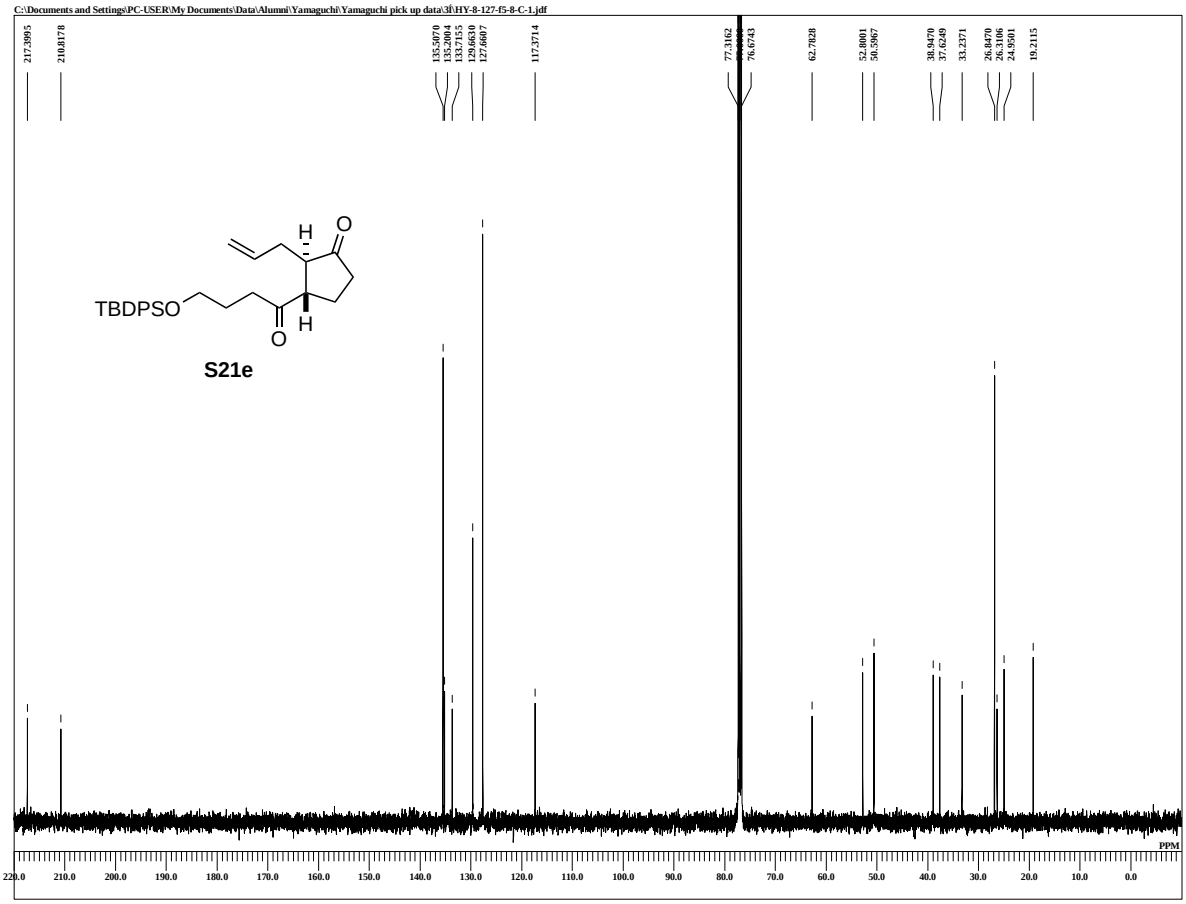
C:\Documents and Settings\PC-USER\My Documents\Data\URABE\HY-8-127-f5-8-1.als



DFILE HY-8-127-f5-8-1.als
COMNT HY-8-127-f5-8
DATIM 08-12-2011 17:38:32
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.62 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 5.0000 sec
SCANS 8
ADBIT 16
RGAIN 40
BF 0.01 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE HY-8-127-f5-8-1.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.9 c
SLVNT CDCL3
EXREF 7.26 ppm

HY-8-127-f5-8-C

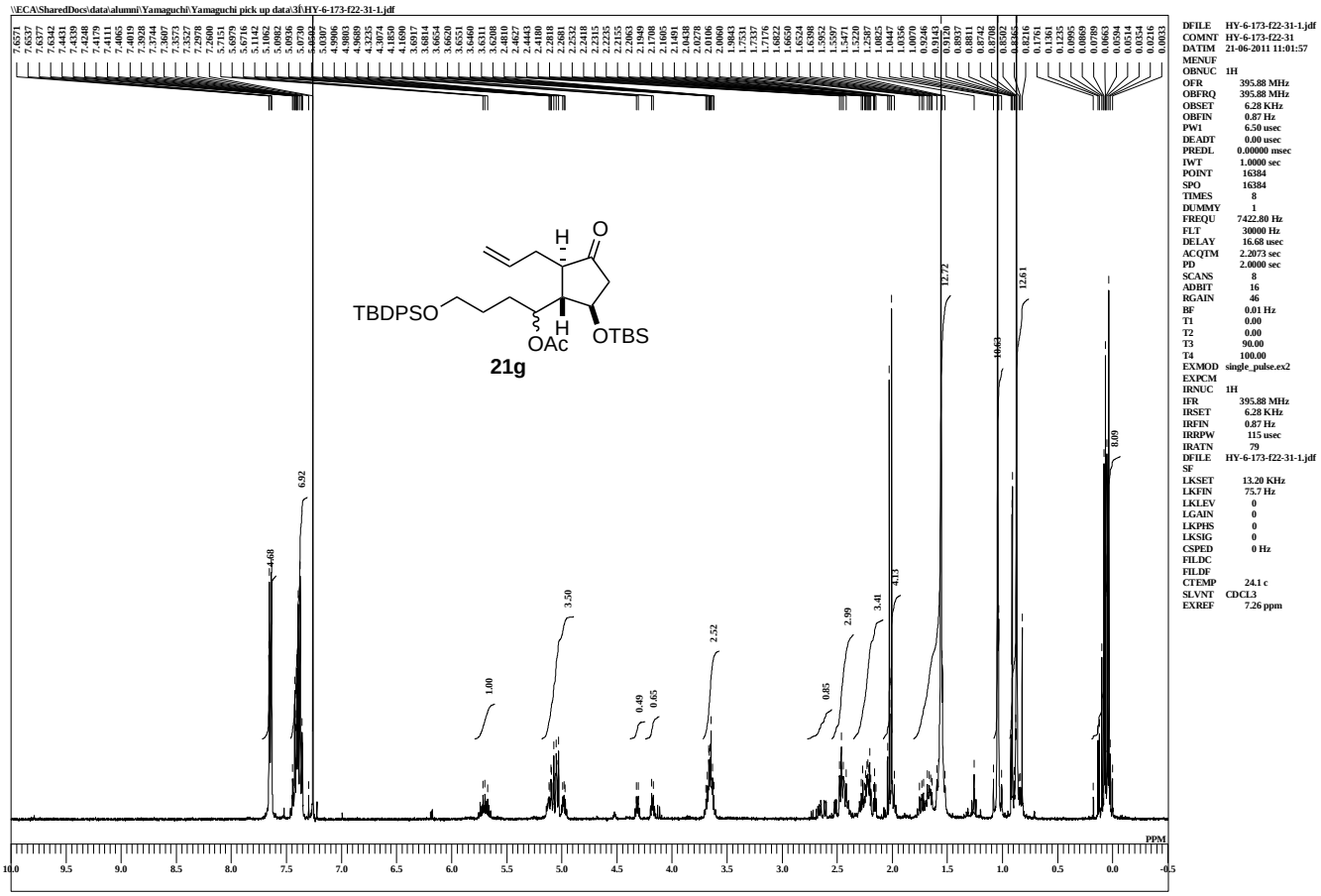
C:\Documents and Settings\PC-USER\My Documents\Data\Alumni\Yamaguchi\Yamaguchi pick up data\3f\HY-8-127-f5-8-C-1.jdf



DFILE HY-8-127-f5-8-C-1.jdf
COMNT HY-8-127-f5-8-C
DATIM 08-12-2011 23:47:18
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 2.92 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 400
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 8.0000 sec
SCANS 400
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE HY-8-127-f5-8-C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.6 c
SLVNT CDCL3
EXREF 77.00 ppm

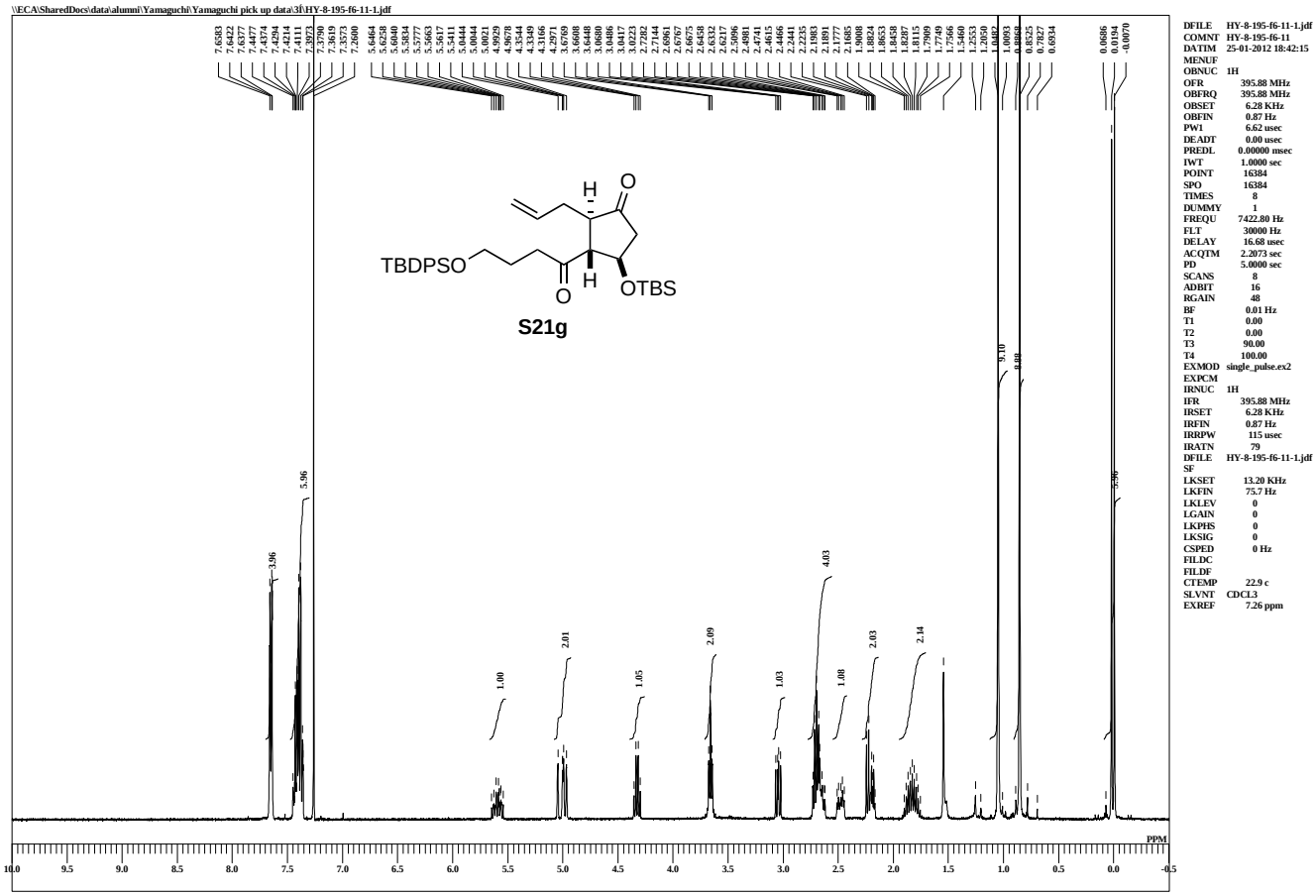
HY-6-173-f22-31

\\ECA\SharedDocs\data\alumni\Yamaguchi\Yamaguchi pick up data\3F\HY-6-173-f22-31-1.jdf

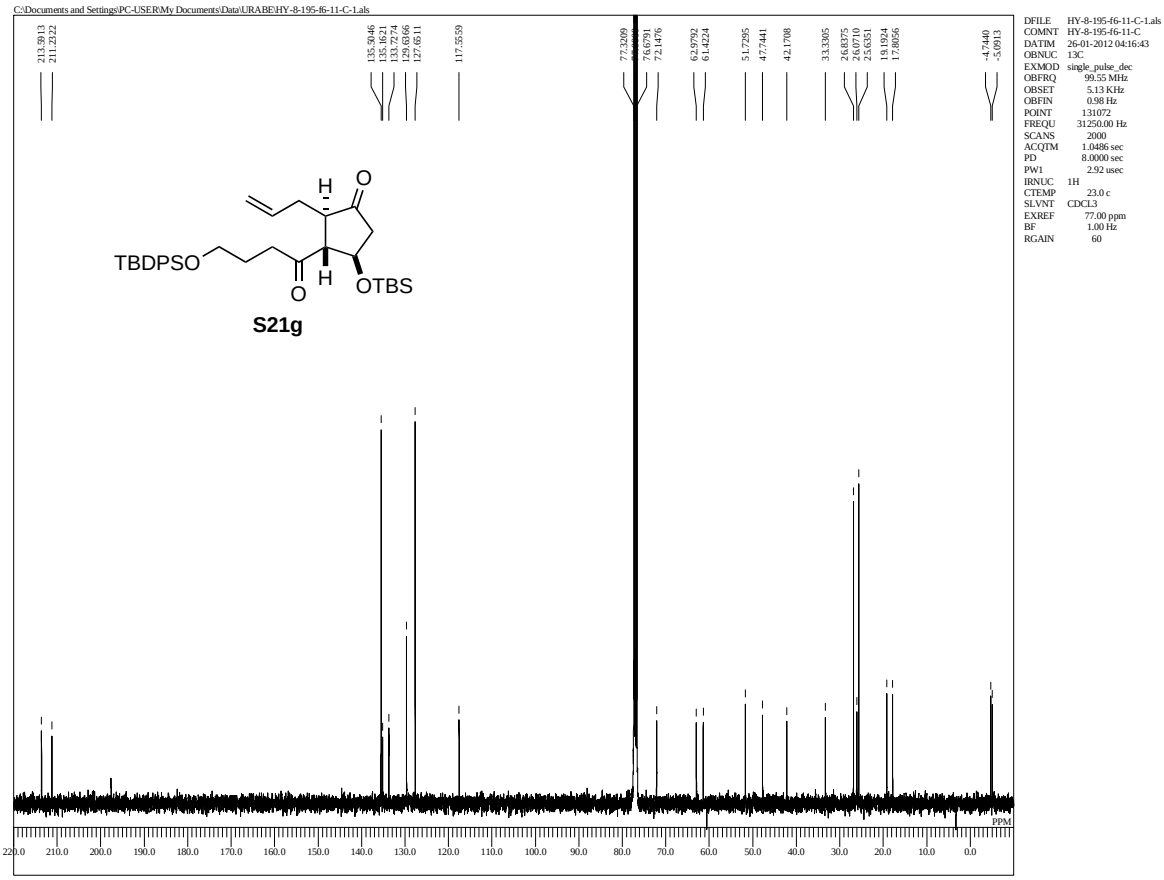


DFILE	HY-6-173-f22-31-1.jdf
COMNT	HY-6-173-f22-31
DATIM	21-06-2011 11:01:57
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.50 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7422.80 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	2.0000 sec
SCANS	8
ADBIT	16
RGAIN	46
BF	0.01 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	HY-6-173-f22-31-1.jdf
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	24.1 c
SLVNT	CDCL3
EXREF	7.26 ppm

HY-8-195-f6-11

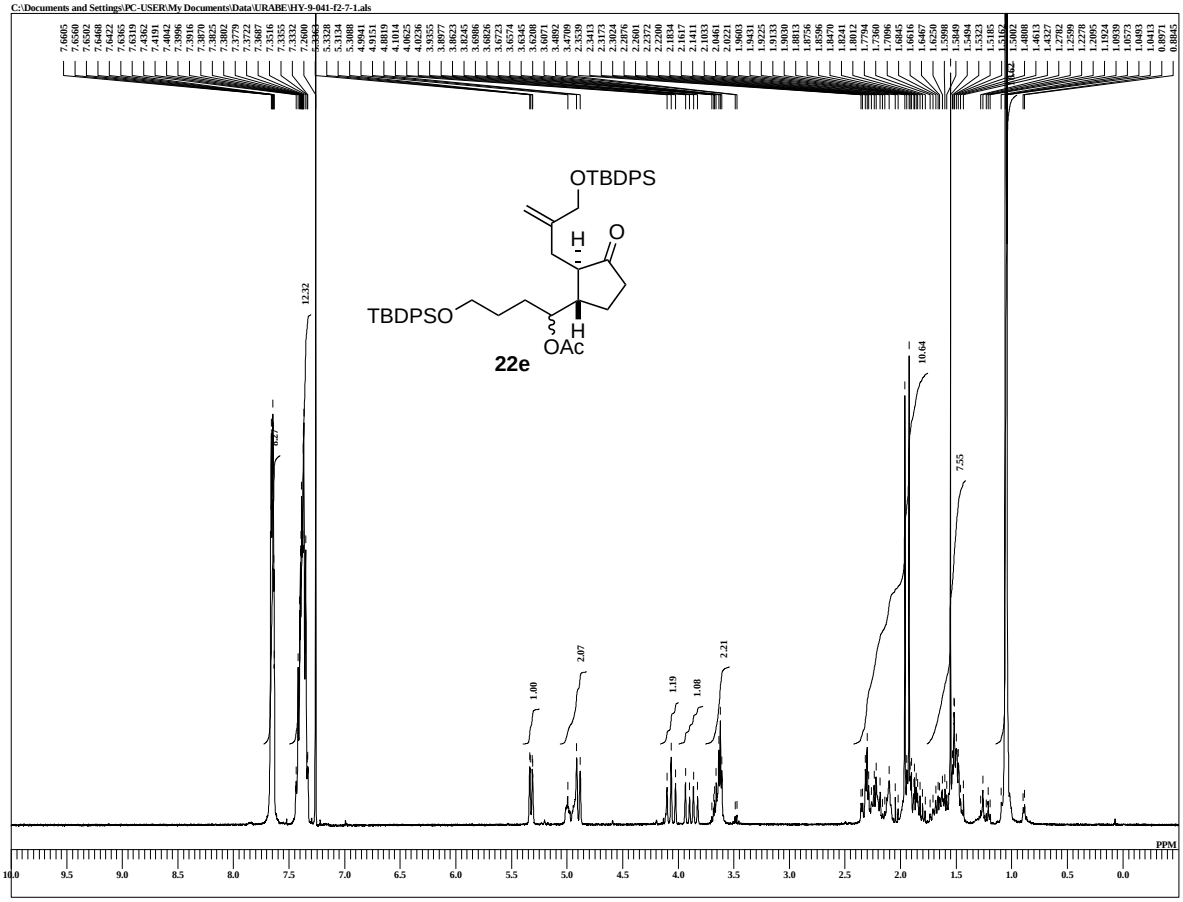


HY-8-195-f6-11-C



HY-9-041-f2-7

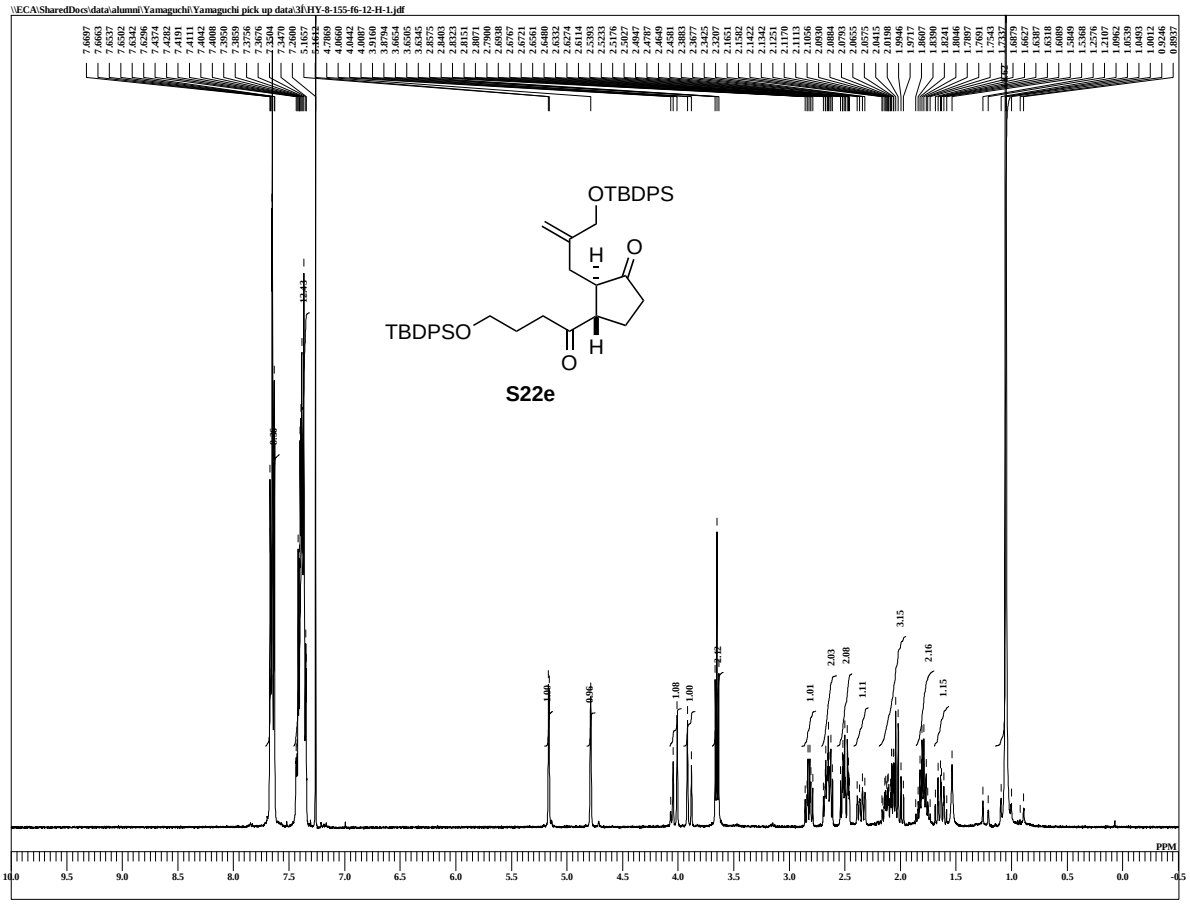
C:\Documents and Settings\PC-USER\My Documents\Data\URABE\HY-9-041-f2-7-1.als



DFILE	HY-9-041-f2-7-1.als
COMNT	HY-9-041-f2-7
DATIM	17-02-2012 21:19:49
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.62 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	13107
SPO	13107
TIMES	16
DUMMY	1
FREQU	5938.15 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	5.0000 sec
SCANS	16
ADBIT	16
RGAIN	44
BF	0.01 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	HY-9-041-f2-7-1.als
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	23.0 c
SLVNT	CDCL3
EXREF	7.26 ppm

HY-8-155-f6-12-H

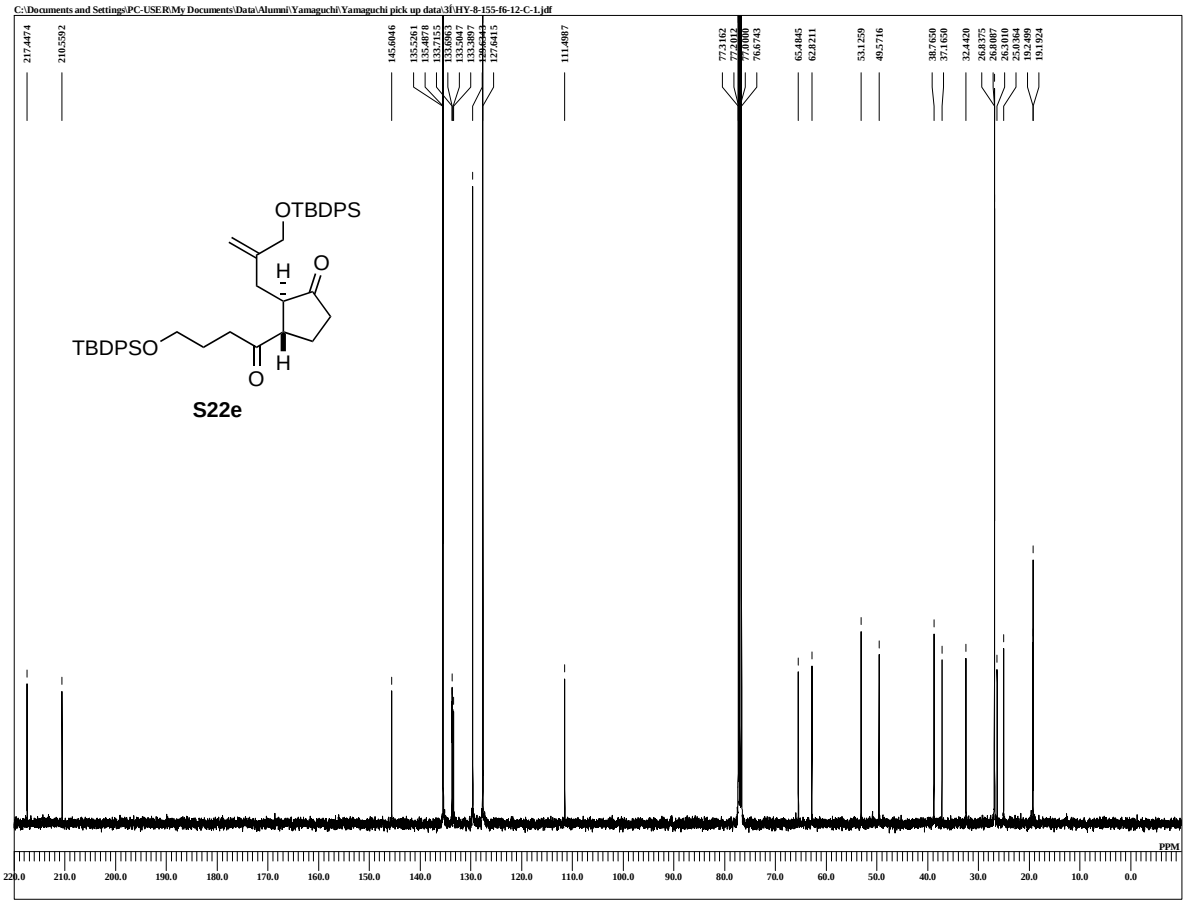
\\ECA\SharedDocs\data\alumni\Yamaguchi\Yamaguchi pick up data\3l\HY-8-155-f6-12-H-1.jdf




```
DFILE    HY-8-155-f6-12-H-1.jdf
COMNT    HY-8-155-f6-12-H
DATIM    24-12-2011 09:16:44
MENUF
OBNUC    1H
OFR      395.88 MHz
OBFRQ    395.88 MHz
OBSET    6.28 KHz
OBFIN    0.87 Hz
PW1      6.62 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    16384
SPO      16384
TIMES    8
DUMMY    1
FREQU    7422.80 Hz
FLT      30000 Hz
DELAY    16.68 usec
ACQTM    2.2073 sec
PD       5.0000 sec
SCANS    8
ADBIT    16
RGAIN    42
BF       0.01 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    HY-8-155-f6-12-H-1.jdf
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    23.0 c
SLVNT    CDCL3
EXREF    7.26 ppm
```

HY-8-155-f6-12-C

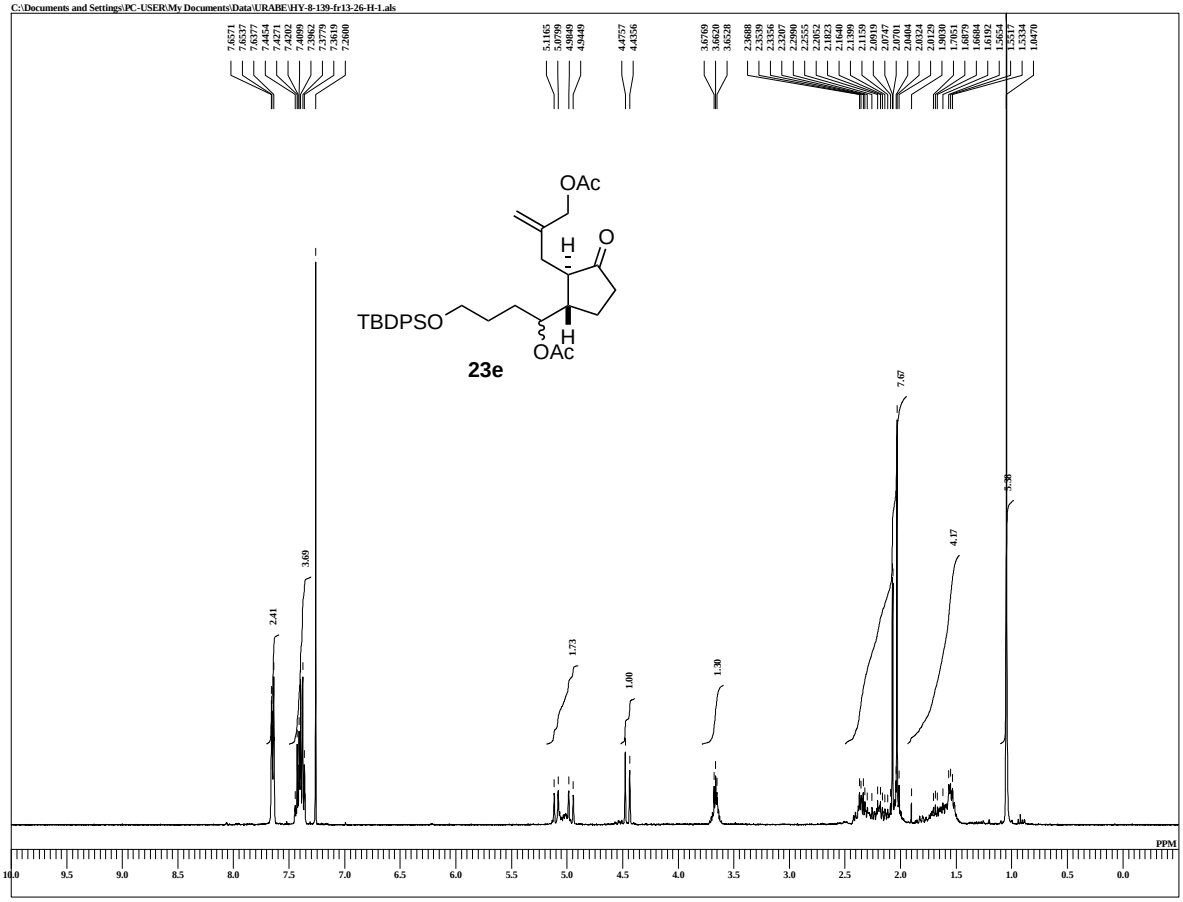
C:\Documents and Settings\PC-USER\My Documents\Data\Alumni\Yamaguchi\Yamaguchi pick up data\3l\HY-8-155-f6-12-C-1.jdf




```
DFILE    HY-8-155-f6-12-C-1.jdf
COMNT    HY-8-155-f6-12-C
DATIM    24-12-2011 10:22:55
MENUF
OBNUC    13C
OFR      99.55 MHz
OBFRQ    99.55 MHz
OBSET    5.13 KHz
OBFIN    0.98 Hz
PW1      2.92 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    32768
SPO      32768
TIMES    400
DUMMY    4
FREQU    31250.00 Hz
FLT      125000 Hz
DELAY    20.50 usec
ACQTM    1.0486 sec
PD       8.0000 sec
SCANS    400
ADBIT    16
RGAIN    60
BF       1.00 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    HY-8-155-f6-12-C-1.jdf
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    23.3 c
SLVNT    CDCL3
EXREF    77.00 ppm
```

HY-8-139-fr13-26-H

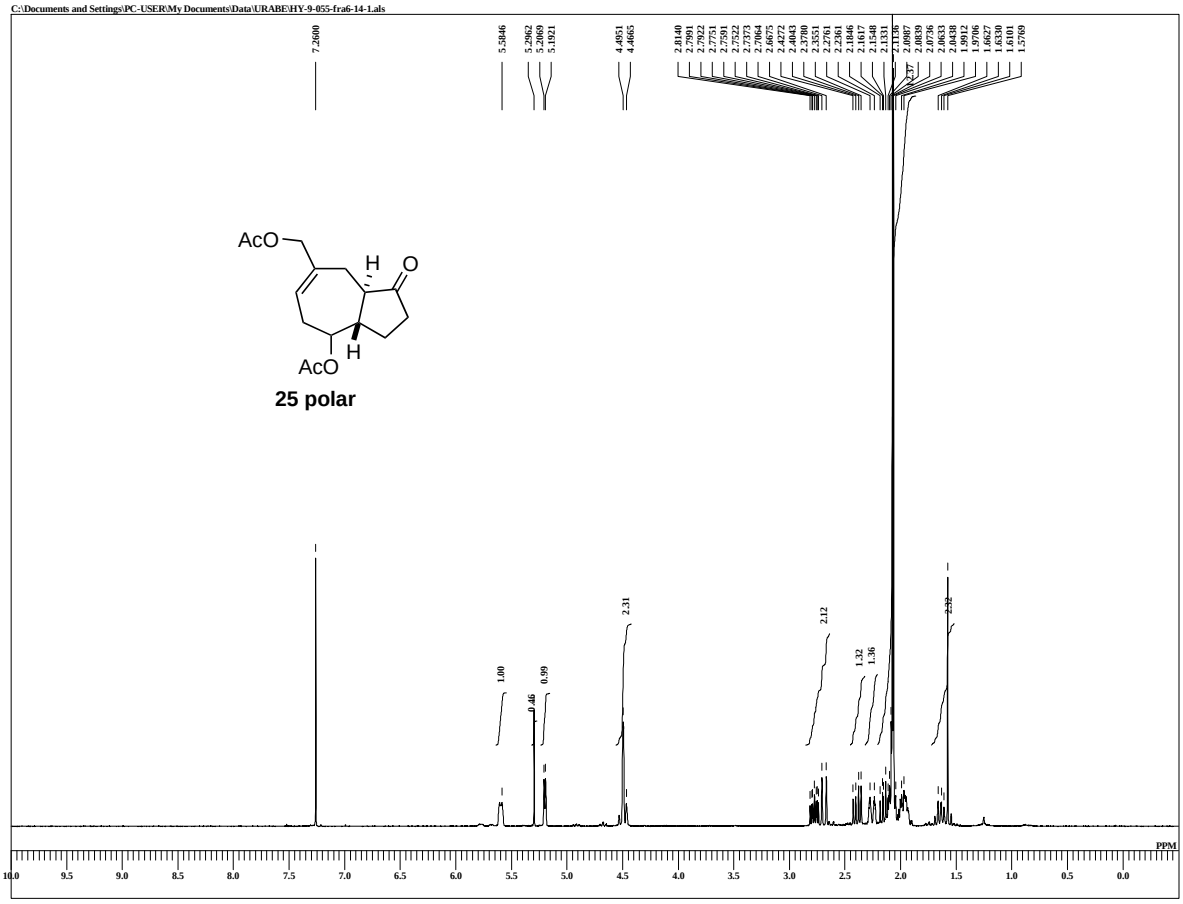
C:\Documents and Settings\PC-USER\My Documents\Data\URABE\HY-8-139-fr13-26-H-1.als



DFILE	HY-8-139-fr13-26-H-1.als
COMNT	HY-8-139-fr13-26-H
DATIM	19-12-2011 16:43:29
MENUF	
OBNUC	1H
OFR	395.88 MHz
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
PW1	6.62 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	13107
SPO	13107
TIMES	8
DUMMY	1
FREQU	5938.15 Hz
FLT	30000 Hz
DELAY	16.68 usec
ACQTM	2.2073 sec
PD	5.0000 sec
SCANS	8
ADBIT	16
RGAIN	44
BF	0.01 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	395.88 MHz
IRSET	6.28 KHz
IRFIN	0.87 Hz
IRRPW	115 usec
IRATN	79
DFILE	HY-8-139-fr13-26-H-1.als
SF	
LKSET	13.20 KHz
LKFIN	75.7 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	
CTEMP	23.1 c
SLVNT	CDCL3
EXREF	7.26 ppm

HY-9-055-fra6-14

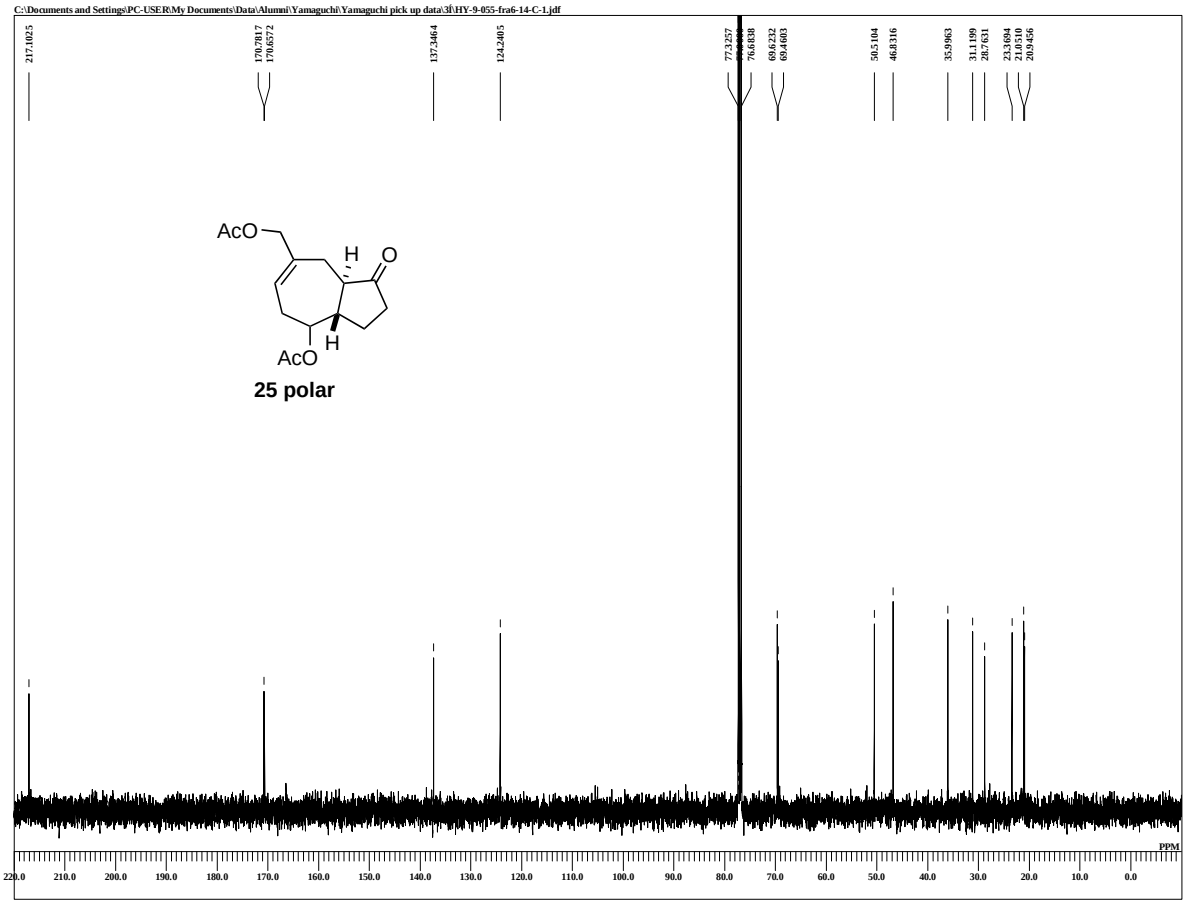
C:\Documents and Settings\PC-USER\My Documents\Data\URABE\HY-9-055-fra6-14-1.als



DFILE HY-9-055-fra6-14-1.als
COMNT HY-9-055-fra6-14
DATIM 01-03-2012 21:20:17
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.62 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 5.0000 sec
SCANS 8
ADBIT 16
RGAIN 44
BF 0.01 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE HY-9-055-fra6-14-1.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 24.3 c
SLVNT CDCL3
EXREF 7.26 ppm

HY-9-055-fra6-14-C

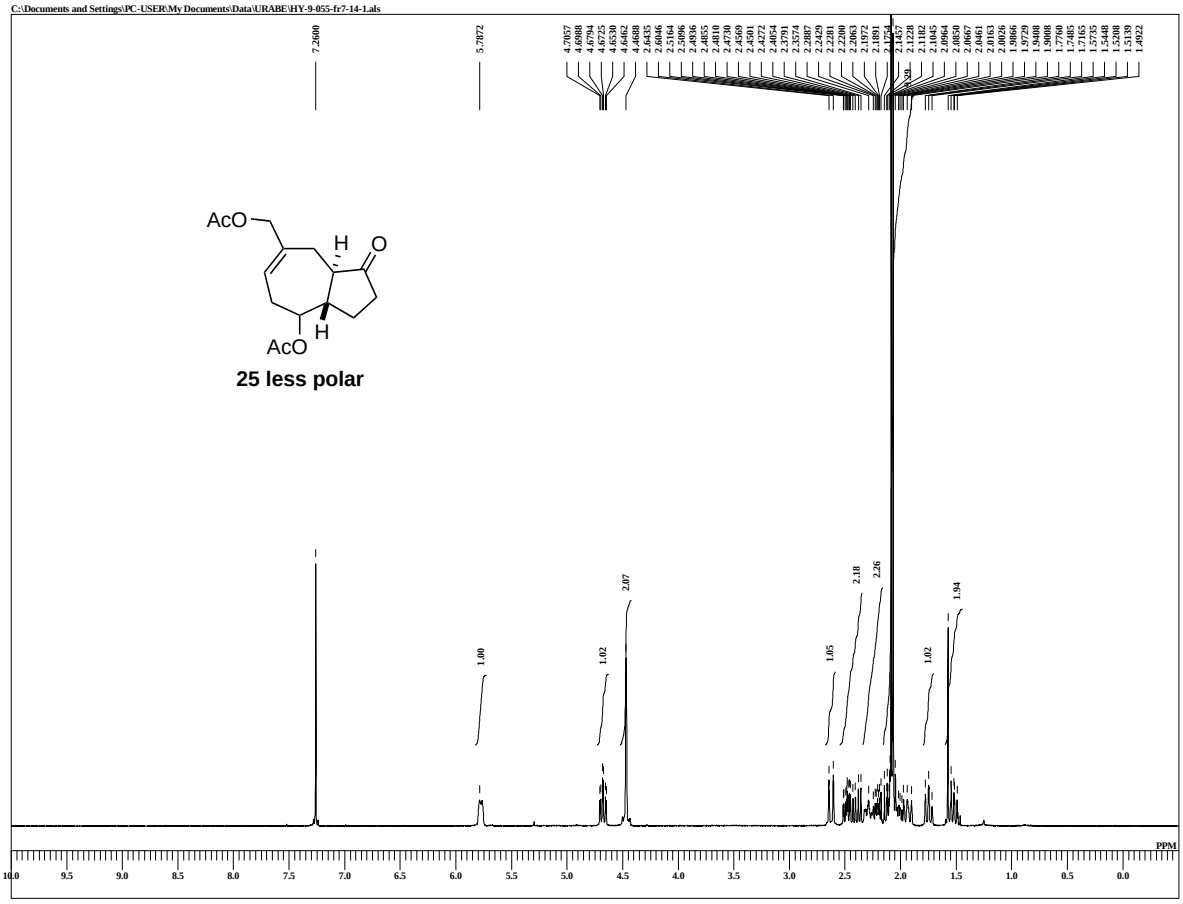
C:\Documents and Settings\PC-USER\My Documents\Data\Alumni\Yamaguchi\Yamaguchi pick up data\3f\HY-9-055-fra6-14-C-1.jdf



DFILE HY-9-055-fra6-14-C-1.jdf
COMNT HY-9-055-fra6-14-C
DATIM 01-03-2012 23:14:02
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 2.92 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 157
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 8.0000 sec
SCANS 157
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE HY-9-055-fra6-14-C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 22.8 c
SLVNT CDCL3
EXREF 77.00 ppm

HY-9-055-fr7-14

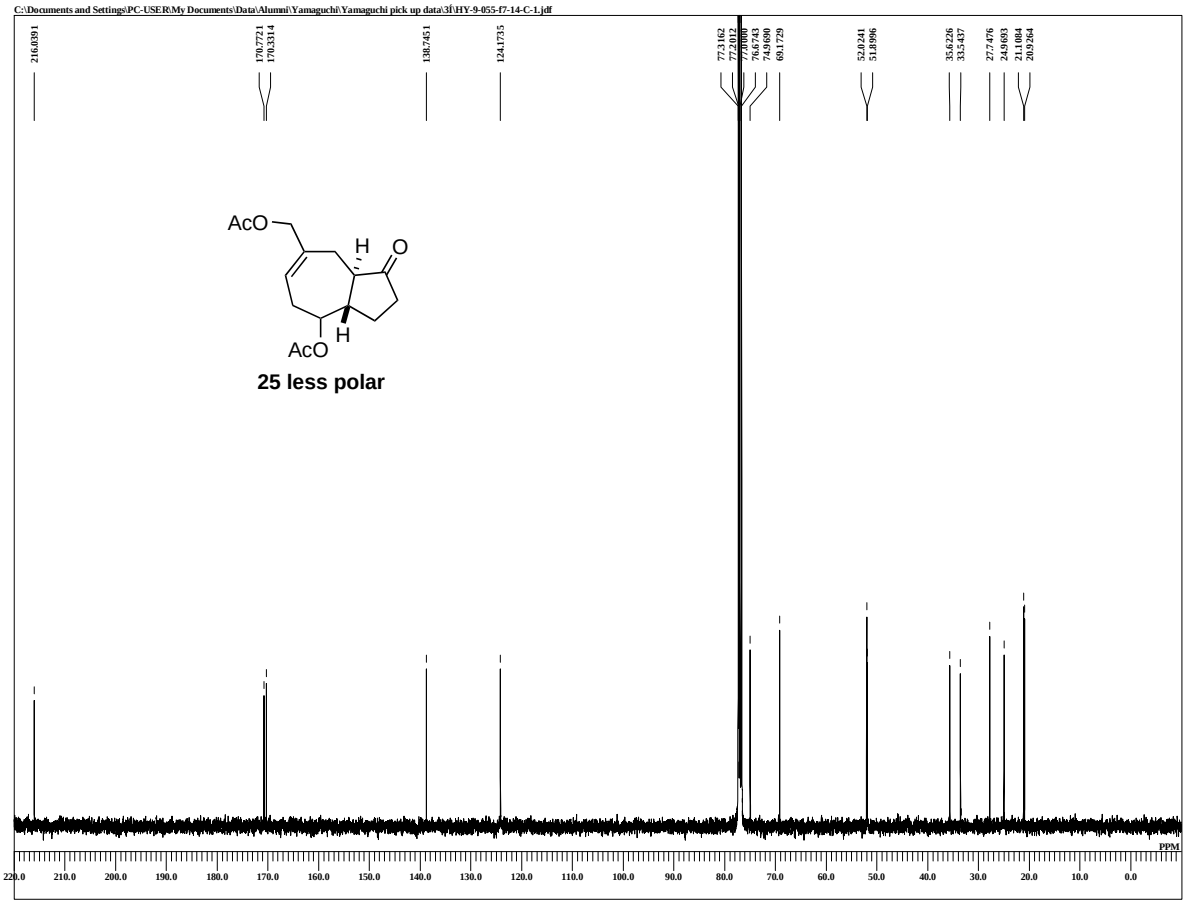
C:\Documents and Settings\PC-USER\My Documents\Data\URABE\HY-9-055-fr7-14-1.als



DFILE HY-9-055-fr7-14-1.als
COMNT HY-9-055-fr7-14
DATIM 01-03-2012 18:06:43
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.62 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 5.0000 sec
SCANS 8
ADBIT 16
RGAIN 44
BF 0.01 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE HY-9-055-fr7-14-1.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 22.8 c
SLVNT CDCL3
EXREF 7.26 ppm

HY-9-055-f7-14-C

C:\Documents and Settings\PC-USER\My Documents\Data\Alumni\Yamaguchi\Yamaguchi pick up data\3f\HY-9-055-f7-14-C-1.jdf



DFILE HY-9-055-f7-14-C-1.jdf
COMNT HY-9-055-f7-14-C
DATIM 02-03-2012 04:08:19
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 2.92 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
SPO 32768
TIMES 1400
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 8.0000 sec
SCANS 1400
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE HY-9-055-f7-14-C-1.jdf
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 22.7 c
SLVNT CDCL3
EXREF 77.00 ppm