Supporting Information

Siloxy(trialkoxy)ethene Undergoes Regioselective [2+2] Cycloaddition to Ynones and Ynoates en route to Functionalized Cyclobutenediones

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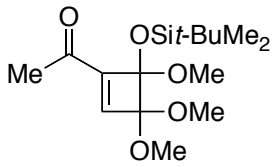
General Experimental Procedures

All experiments dealing with air- and moisture-sensitive compounds were conducted under an atmosphere of dry argon.

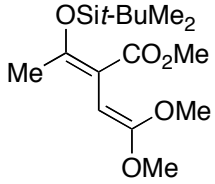
For thin-layer chromatography (TLC) analysis, Merck pre-coated plates (silica gel 60 F_{254}, Art 5715, 0.25 mm) were used. For flash column chromatography, silica gel 60 (Merck Art 7734, 70–230 mesh) was used. Silica gel preparative TLC (PTLC) was performed on Merck silica gel 60 PF_{254} (Art 7747).

Melting point (mp) determinations were performed by using a Yanako MP-S3 instrument and are uncorrected. ^{1}H NMR and ^{13}C NMR were measured on a JEOL JNM lambda-400, a JEOL JNM ECA-400, and a Bruker AV400N spectrometer. Infrared (IR) spectra were recorded on a Jasco IR-Report-100, and a Horiba FT-710 spectrometer. Attenuated Total Reflectance Fourier Transformation Infrared (ATR-FTIR) spectra were recorded on a Perkin Elmer 1600 FTIR. Elementary analyses were performed by using Perkin Elmer series II 2004.

*Synthesis of cyclobutene **3**:*
A mixture of 3-butyne-2-one (**1**) (131 mg, 1.92 mmol) and KSA **2** (627 mg, 2.52 mmol) was heated at 60 °C for 8 h. The crude product was purified by silica-gel flash column chromatography (hexane/EtOAc = 9/1) to give **3** (564 mg, 92.6%) as a colorless oil.

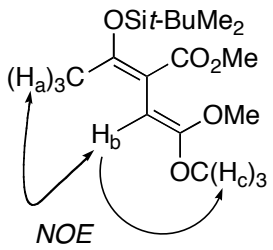


cyclobutene **3**
^{1}H NMR (acetone-d_6, δ)
0.08 (s, 3H), 0.14 (s, 3H), 0.90 (s, 9H), 2.29 (s, 3H), 3.33 (s, 3H), 3.36 (s, 3H), 3.39 (s, 3H), 7.33 (s, 1H);
^{13}C NMR (acetone-d_6, δ)
–3.4, –3.2, 19.1, 26.3, 27.5, 51.2, 51.3, 53.1, 107.0, 108.3, 145.8, 152.3, 194.9;
IR (neat)
2937, 2856, 1689, 1606, 1463, 1361, 1276, 1251, 1195, 1095, 1014, 998, 898, 838, 781 cm^{-1};
Anal. Calcd for $C_{15}H_{28}O_5Si$: C, 56.93; H, 8.92. Found: C, 56.77; H, 9.01.



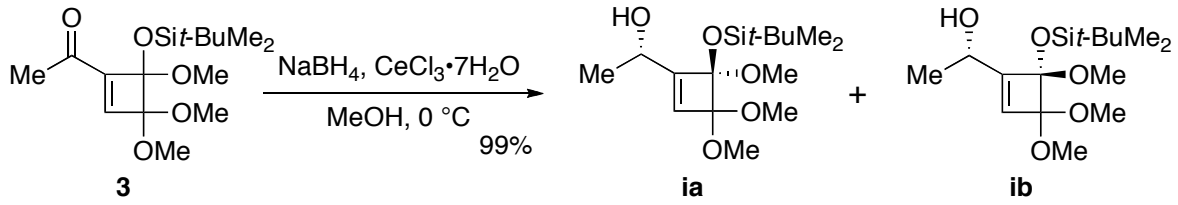
1,3-diene **4**
^{1}H NMR (acetone-d_6, δ)
0.15 (s, 6H), 0.91 (s, 9H), 1.86 (s, 3H), 3.49 (s, 3H), 3.60 (s, 3H), 3.63 (s, 3H), 4.30 (s, 1H);
^{13}C NMR (acetone-d_6, δ)
–3.7, 18.6, 20.0, 26.0, 51.1, 55.6, 56.1, 76.3, 111.8, 148.8, 159.7, 169.3;
IR (neat)
2950, 2897, 2858, 1734, 1667, 1619, 1462, 1434, 1381, 1324, 1254, 1213, 1169, 1104, 1076, 1002, 939, 863, 838, 813, 780 cm^{-1}.

[#1]The stereochemistry of **4** was determined on the basis of the observed NOE shown below.



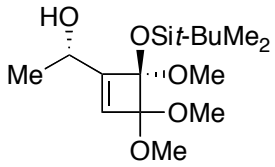
H$_a$: 1.86 ppm, H$_b$: 4.30 ppm, H$_c$: 3.63 ppm

Scheme 1. Determination of the regiochemistry



3 **ia** **ib**

*Synthesis of alcohol **i**:*
To a solution of cyclobutene **3** (33.5 mg, 0.106 mmol) in MeOH (0.3 mL) was added $CeCl_3$•$7H_2O$ (43.5 mg, 0.117 mmol) and $NaBH_4$ (4.5 mg, 0.119 mmol) at 0 °C. After 1 h, the reaction was stopped by adding water. The products were extracted with Et_2O (X3), and the combined organic extracts were washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by PTLC (hexane/CH_2Cl_2/Et_2O = 50/25/25) to give alcohol **i** (33.3 mg, 98.8%) as a mixturer of diasteromers.
Recrystallization from hexane gave **ia** as colorless prisms. Mp 72.0–73.5 °C.



alcohol **ia**
^{1}H NMR (acetone-d_6, δ)
0.149 (s, 3H), 0.154 (s, 3H), 0.90 (s, 9H), 1.32 (d, 3H, J = 6.8 Hz), 3.27 (s, 3H), 3.30 (s, 3H), 3.41 (s, 3H), 3.89 (d, 1H, J = 5.3 Hz), 4.40–4.50 (m, 1H), 6.47 (d, 1H, J = 1.4 Hz);
^{13}C NMR (acetone-d_6, δ)
–2.9, –2.7, 19.0, 22.8, 26.5, 50.87, 50.91, 53.2, 64.2, 107.7, 108.3, 131.4, 161.6;
IR (ATR)
3501, 2938, 2893, 2857, 1467, 1436, 1387, 1359, 1333, 1277, 1243, 1193, 1165, 1080, 1058, 1009, 982, 905, 829, 780, 727 cm^{-1};
Anal. Calcd for $C_{15}H_{30}O_5Si$: C, 56.57; H, 9.49. Found: C, 56.65; H, 9.42.

[#2] The stereochemistry of alcohol **ia** was determined by X-ray analysis shown below.[1]

[1] CCDC-662792 (**ia**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

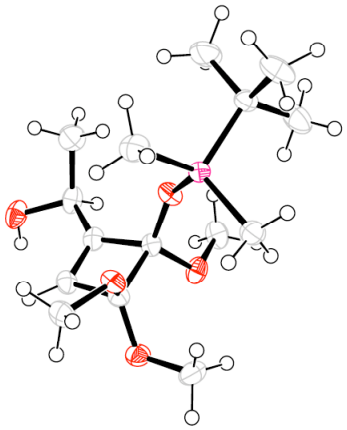
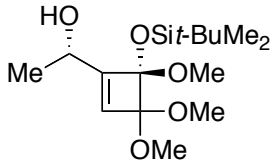
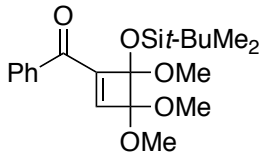


Figure 1. X-ray structure of **ia**



alcohol **ib**
^{1}H NMR (acetone-d_6, δ)
0.14 (s, 3H), 0.14 (s, 3H), 0.89 (s, 9H), 1.29 (d, 3H, J = 6.6 Hz), 3.26 (s, 3H), 3.30 (s, 3H), 3.40 (s, 3H), 3.95 (d, 1H, J = 5.1 Hz), 4.40–4.50 (m, 1H), 6.44 (d, 1H, J = 1.4 Hz);
^{13}C NMR (acetone-d_6, δ)
–3.2, –2.9, 19.1, 23.0, 26.3, 51.0, 51.2, 52.3, 63.9, 106.9, 109.0, 130.7, 161.7;
IR (neat)
3454, 2952, 2935, 2856, 2832, 1473, 1463, 1438, 1373, 1361, 1276, 1253, 1193, 1164, 1089, 1058, 1018, 989, 921, 894, 836, 779, 730 cm^{-1}
Anal. Calcd for $C_{15}H_{30}O_5Si$: C, 56.57; H, 9.49. Found: C, 56.80; H, 9.62.

*Synthesis of cyclobutene **6a**:*
According to the general procedure for synthesis of cyclobutene **3**, ynone **5a**[2] (124 mg, 0.953 mmol) and KSA **2** (318 mg, 1.28 mmol) gave, after purification by silica-gel flash column chromatography (hexane/EtOAc = 9/1), **6a** (337 mg, 93.4%) as a yellow oil.

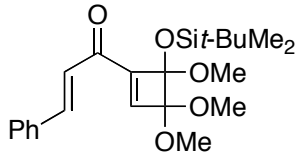


cyclobutene **6a**
^{1}H NMR (acetone-d_6, δ)
0.08 (s, 3H), 0.16 (s, 3H), 0.89 (s, 9H), 3.39 (s, 3H), 3.42 (s, 3H), 3.47 (s, 3H), 7.21 (s, 1H), 7.52–7.60 (m, 2H), 7.64–7.70 (m, 1H), 7.93–7.98 (m, 2H);
^{13}C NMR (acetone-d_6, δ)

[2] Y. Maeda, N. Kakiuchi, S. Matsumura, T. Nishimura, T. Kawamura, S. Uemura, *J. Org. Chem.* **2002**, *67*, 6718.

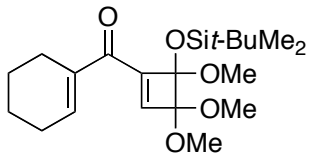
–3.2, –3.1, 19.0, 26.3, 51.3, 51.4, 53.4, 108.2, 108.9, 129.6, 129.8, 134.2, 137.9, 145.3, 151.3, 190.4;
IR (neat)
3066, 2937, 2904, 2856, 2835, 1660, 1598, 1580, 1472, 1463, 1448, 1360, 1318, 1278, 1256, 1193, 1150, 1107, 1090, 1065, 1013, 1000, 941, 892, 874, 839, 801, 781, 721 cm^{-1};
Anal. Calcd for $C_{20}H_{30}O_5Si$: C, 63.46; H, 7.99. Found: C, 63.34; H, 8.00.

*Synthesis of cyclobutene **6b**:*
According to the general procedure for synthesis of cyclobutene **3**, ynone **5b**[2] (78.3 mg, 0.501 mmol) and KSA **2** (178 mg, 0.717 mmol) gave, after purification by silica-gel flash column chromatography (hexane/EtOAc = 9/1), **6b** (180 mg, 88.8%). Recrystallization from hexane gave **6b** as yellow prisms. Mp. 68.0–69.2 °C.



cyclobutene **6b**
^{1}H NMR (acetone-d_6, δ)
0.13 (s, 3H), 0.18 (s, 3H), 0.93 (s, 9H), 3.38 (s, 3H), 3.41 (s, 3H), 3.48 (s, 3H), 7.41 (d, 1H, J = 15.8 Hz), 7.45–7.50 (m, 3H), 7.54 (s, 1H), 7.71 (d, 1H, J = 15.8 Hz), 7.75–7.82 (m, 2H);
^{13}C NMR (acetone-d_6, δ)
–3.2, –3.1, 19.1, 26.4, 51.3, 51.4, 53.3, 107.2, 108.6, 124.0, 129.5, 129.8, 131.6, 135.6, 144.3, 145.3, 153.2, 186.2;
IR (ATR)
3062, 3026, 2937, 2903, 2856, 2834, 1666, 1636, 1597, 1576, 1495, 1471, 1462, 1450, 1408, 1388, 1339, 1277, 1180, 1093, 1066, 1010, 999, 912, 887, 838, 796, 781, 766, 727 cm^{-1};
Anal. Calcd for $C_{22}H_{32}O_5Si$: C, 65.31; H, 7.97. Found: C, 65.51; H, 8.14.

*Synthesis of cyclobutene **6c**:*
According to the general procedure for synthesis of cyclobutene **3**, ynone **5c**[3] (53.5 mg, 0.399 mmol) and KSA **2** (141 mg, 0.568 mmol) gave, after purification by PTLC (hexane/EtOAc = 7/3), **6c** (115 mg, 75.4%) as a colorless oil.

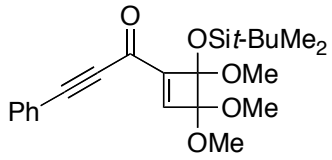


cyclobutene **6c**
^{1}H NMR (acetone-d_6, δ)
0.05 (s, 3H), 0.13 (s, 3H), 0.89 (s, 9H), 1.58–1.70 (m, 4H), 2.16–2.36 (m, 4H), 3.34 (s, 3H), 3.36 (s, 3H), 3.43 (s, 3H), 6.93 (s, 1H), 7.08–7.12 (m, 1H);
^{13}C NMR (acetone-d_6, δ)
–3.14, –3.11, 18.9, 22.3, 22.5, 23.5, 26.3, 26.7, 51.16, 51.23, 53.4, 108.5, 108.7, 139.9, 141.8, 145.3, 152.0, 191.4;

[3] E. Schroeder, M. Lehmann, I. Boettcher, *Eur. J. Med. Chem.* **1979**, *14*, 309.

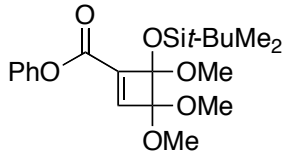
IR (neat)
3068, 2935, 2857, 2834, 1647, 1637, 1472, 1460, 1449, 1437, 1422, 1388, 1378, 1360, 1343, 1306, 1278, 1248, 1192, 1139, 1099, 1064, 1013, 998, 971, 927, 889, 838, 799, 781, 749, 706 cm^{-1};
Anal. Calcd for $C_{20}H_{34}O_5Si$: C, 62.79; H, 8.96. Found: C, 63.02; H, 8.75.

*Synthesis of cyclobutene **6d**:*
According to the general procedure for synthesis of cyclobutene **3**, ynone **5d**[4] (148 mg, 0.960 mmol) and KSA **2** (323 mg, 1.30 mmol) gave, after purification by silica-gel flash column chromatography (hexane/EtOAc = 19/1), **6d** (377 mg, 97.6%) as a colorless oil.



cyclobutene **6d**
^{1}H NMR (acetone-d_6, δ)
0.13 (s, 3H), 0.17 (s, 3H), 0.91 (s, 9H), 3.38 (s, 3H), 3.41 (s, 3H), 3.45 (s, 3H), 7.45–7.65 (m, 3H), 7.69 (s, 1H), 7.72–7.80 (m, 2H);
^{13}C NMR (acetone-d_6, δ)
–3.4, –3.3, 19.1, 26.2, 51.3, 51.5, 53.4, 86.9, 92.8, 106.6, 108.1, 120.3, 129.8, 132.2, 134.1, 149.9, 152.8, 173.3;
IR (neat)
3065, 2938, 2856, 2836, 2200, 1644, 1597, 1490, 1471, 1463, 1443, 1318, 1273, 1190, 1093, 1009, 896, 838, 798, 781, 758, 688 cm^{-1};
Anal. Calcd for $C_{22}H_{30}O_5Si$: C, 65.64; H, 7.51. Found: C, 65.42; H, 7.72.

*Synthesis of cyclobutene **6e**:*
According to the general procedure for synthesis of cyclobutene **3**, ynoate **5e**[5] (40.0 mg, 0.273 mmol) and KSA **2** (89.5 mg, 0.360 mmol) gave, after purification by PTLC (hexane/EtOAc = 9/1), **6e** (101 mg, 93.5%) as a colorless oil.

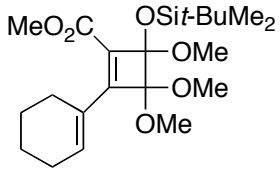


cyclobutene **6e**
^{1}H NMR (acetone-d_6, δ)
0.17 (s, 3H), 0.19 (s, 3H), 0.93 (s, 9H), 3.38 (s, 3H), 3.41 (s, 3H), 3.51 (s, 3H), 7.17–7.21 (m, 2H), 7.25–7.32 (m, 1H), 7.42–7.48 (m, 2H), 7.54 (s, 1H);
^{13}C NMR (acetone-d_6, δ)
–3.3, –3.1, 19.0, 26.2, 51.3, 51.4, 53.0, 106.9, 108.6, 122.4, 126.9, 130.4, 145.8, 149.1, 151.2, 160.5;
IR (neat)

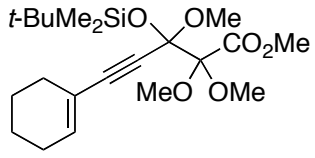
[4] D. H. Wadsworth, S. M. Geer, M. R. Detty, *J. Org. Chem.* **1987**, *52*, 3662.
[5] P. V. Ramachandran, M. T. Rudd, M. V. R. Reddy, *Tetrahedron Lett.* **2005**, *46*, 2547.
[6] L. G. Beholz, P. Benovsky, D. L. Ward, N. S. Barta, J. R. Stille, *J. Org. Chem.* **1997**, *62*, 1033.

3075, 3044, 2938, 2897, 2857, 2836, 1744, 1619, 1591, 1492, 1472, 1463, 1409, 1389, 1360, 1275, 1239, 1192, 1162, 1094, 1071, 1052, 1012, 902, 868, 839, 798, 781, 756, 733 cm^{-1};
Anal. Calcd for $C_{20}H_{30}O_6Si$: C, 60.89; H, 7.66. Found: C, 60.65; H, 7.79.

*Synthesis of cyclobutene **8a**:*
To a solution of ynoate **7a**[5] (80.1 mg, 0.488 mmol) in toluene (1.0 mL) was added Me_3Al (1.03 M in hexane, 0.48 mL, 0.49 mmol) and KSA **2** (141 mg, 0.567 mmol) in toluene (1.0 mL) at –78 °C. After warmed up to –20 °C and further stirred for further 69 h, the reaction was stopped by adding sat. aq. $NaHCO_3$. The products were extracted with EtOAc and combined organic extracts were washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by PTLC (hexane/CH_2Cl_2/Et_2O = 70/15/15) to give cyclobutene **8a** (89.4 mg, 44.4%) and adduct **9a** (57.5 mg, 28.6%).



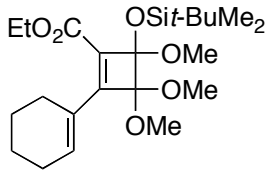
cyclobutene **8a** (colorless oil)
^{1}H NMR (acetone-d_6, δ)
0.10 (s, 3H), 0.14 (s, 3H), 0.91 (s, 9H), 1.55–1.67 (m, 4H), 2.14–2.43 (m, 4H), 3.40 (s, 3H), 3.41 (s, 3H), 3.42 (s, 3H), 3.73 (s, 3H), 6.58–6.61 (m, 1H);
^{13}C NMR (acetone-d_6, δ)
–3.1, –3.0, 19.0, 22.2, 23.0, 26.3, 26.5, 26.9, 51.4, 52.4, 52.5, 52.6, 106.7, 109.2, 132.8, 133.5, 137.5, 156.8, 163.6;
IR (neat)
2936, 2857, 1720, 1627, 1461, 1434, 1268, 1245, 1218, 1199, 1087, 1025, 998, 952, 890, 836, 802, 779, 728 cm^{-1};
Anal. Calcd for $C_{21}H_{36}O_6Si$: C, 61.13; H, 8.79. Found: C, 61.11; H, 8.95.



adduct **9a** (colorless oil)
^{1}H NMR (acetone-d_6, δ)
0.205 (s, 3H), 0.211 (s, 3H), 0.90 (s, 9H), 1.56–1.67 (m, 4H), 2.10–2.12 (m, 4H), 3.381 (s, 3H), 3.383 (s, 3H), 3.40 (s, 3H), 3.65 (s, 3H), 6.10–6.18 (m, 1H);
^{13}C NMR (acetone-d_6, δ)
–3.4, –2.6, 18.8, 22.0, 22.8, 26.08, 26.13, 29.3, 51.85, 51.92, 52.01, 52.02, 84.2, 90.1, 97.3, 104.3, 120.6, 136.5, 167.5;
IR (neat)
2935, 2857, 2217, 1751, 1461, 1434, 1388, 1361, 1284, 1249, 1214, 1126, 1072, 1018, 960, 937, 879, 840, 782, 717 cm^{-1};
Anal. Calcd for $C_{21}H_{36}O_6Si$: C, 61.13; H, 8.79. Found: C, 61.23; H, 8.64.

*Synthesis of cyclobutene **8b**:*

According to the general procedure for synthesis of **8a**, ynoate **7b**[5] (90.1 mg, 0.506 mmol), KSA **2** (147 mg, 0.592 mmol), and Me_3Al (1.03 M in hexane, 0.50 mL, 0.51 mmol) gave, after purification by PTLC (hexane/EtOAc = 9/1), cyclobutene **8b** (105 mg, 48.7%) and adduct **9b** (61.8 mg, 28.7%).



cyclobutene **8b** (color less oil)

^{1}H NMR (acetone-d_6, δ)

0.12 (s, 3H), 0.15 (s, 3H), 0.91 (s, 9H), 1.28 (t, 3H, J = 7.0 Hz), 1.55–1.70 (m, 4H), 2.14–2.24 (m, 2H), 2.24–2.42 (m, 2H), 3.400 (s, 3H), 3.403 (s, 3H), 3.43 (s, 3H), 4.17 (qd, 1H, J_1 = 7.0, J_2 = 11.1 Hz), 4.24 (qd, 1H, J_1 = 7.0, J_2 = 11.1 Hz), 6.55–6.61 (m, 1H);

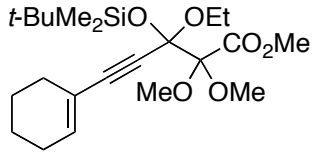
^{13}C NMR (acetone-d_6, δ)

–3.0, –2.9, 14.5, 19.0, 22.2, 23.0, 26.3, 26.5, 27.0, 52.2, 52.6, 52.8, 60.9, 106.8, 109.1, 132.0, 134.1, 137.1, 156.4, 163.4;

IR (neat)

2935, 2857, 2834, 1716, 1627, 1461, 1446, 1388, 1365, 1307, 1268, 1245, 1218, 1191, 1091, 1029, 998, 952, 894, 836, 802, 779, 725 cm^{-1};

Anal. Calcd for $C_{22}H_{38}O_6Si$: C, 61.94; H, 8.98. Found: C, 61.71; H, 9.16.



adduct **9b** (colorless oil)

^{1}H NMR (acetone-d_6, δ)

0.18 (s, 3H), 0.20 (s, 3H), 0.90 (s, 9H), 1.11 (t, 3H, J = 7.0 Hz), 1.55–1.70 (m, 4H), 2.08–2.14 (m, 4H), 3.38 (s, 3H), 3.41 (s, 3H), 3.66 (s, 3H), 3.75 (q, 1H, J = 7.0 Hz), 3.75 (q, 1H, J = 7.0 Hz), 6.08–6.16 (m, 1H);

^{13}C NMR (acetone-d_6, δ)

–3.4, –2.5, 15.6, 18.7, 22.0, 22.8, 26.07, 26.11, 29.3, 51.85, 51.88, 61.1, 84.8, 89.9, 96.9, 104.6, 120.8, 136.3, 167.7;

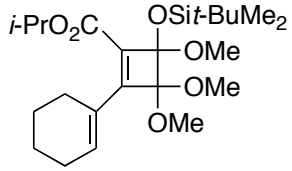
IR (neat)

2931, 2857, 2217, 1751, 1461, 1434, 1388, 1361, 1284, 1249, 1211, 1137, 1106, 1068, 1018, 937, 890, 840, 779, 721 cm^{-1};

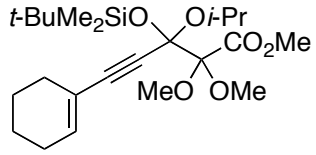
Anal. Calcd for $C_{22}H_{38}O_6Si$: C, 61.94; H, 8.98. Found: C, 61.70; H, 9.16.

*Synthesis of cyclobutene **8c**:*

According to the general procedure for synthesis of cyclobutene **8a**, ynoate **7c** (47.0 mg, 0.244 mmol), KSA **2** (72.0 mg, 0.290 mmol), and Me_3Al (1.02 M in hexane, 0.24 mL, 0.24 mmol) gave, after purification by PTLC (hexane/EtOAc = 9/1), cyclobutene **8c** (63.9 mg, 59.3%) and adduct **9c** (10.8 mg, 10.0%).

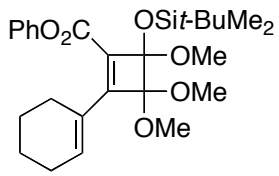


cyclobutene **8c** (colorless oil)
^{1}H NMR (acetone-d_6, δ)
0.14 (s, 3H), 0.16 (s, 3H), 0.91 (s, 9H), 1.27 (d, 3H, J = 6.4 Hz), 1.28 (d, 3H, J = 6.4 Hz), 1.54–1.70 (m, 4H), 2.15–2.22 (m, 2H), 2.28–2.37 (m, 2H), 3.397 (s, 3H), 3.401 (s, 3H), 3.43 (s, 3H), 5.08 (sept, 1H, J = 6.4 Hz), 6.54–6.57 (m, 1H);
^{13}C NMR (acetone-d_6, δ)
–2.9, –2.8, 19.0, 21.9, 22.0, 22.2, 23.0, 26.4, 26.4, 27.1 52.1 52.6, 52.9, 68.7, 106.9, 109.0, 131.9, 134.5, 136.7, 155.9, 163.0;
IR (neat)
2935, 2857, 2838, 1712, 1627, 1465, 1384, 1357, 1307, 1268, 1249, 1222, 1195, 1091, 1022, 998, 952, 921, 890, 836, 806, 779, 725 cm^{-1};
Anal. Calcd for $C_{23}H_{40}O_6Si$: C, 62.69; H, 9.15. Found: C, 62.68; H, 9.08.



adduct **9c** (colorless oil)
^{1}H NMR (acetone-d_6, δ)
0.23 (s, 3H), 0.24 (s, 3H), 0.90 (s, 9H), 1.16 (d, 6H, J = 6.4 Hz), 1.54–1.70 (m, 4H), 2.06–2.15 (m, 4H), 3.407 (s, 3H), 3.410 (s, 3H), 3.65 (s, 3H), 4.28 (sept, 1H, J = 6.4 Hz), 6.10–6.17 (m, 1H);
^{13}C NMR (acetone-d_6, δ)
–3.1, –2.4, 18.8, 22.0, 22.7, 23.5, 24.7, 26.0, 26.2, 51.7 51.9, 52.3, 68.1, 85.6, 88.7, 96.6, 104.1, 120.7, 136.2, 167.8;
IR (neat)
2935, 2857, 2217, 1751, 1627, 1465, 1434, 1380, 1361, 1284, 1249, 1214, 1114, 1064, 1041, 956, 917, 887, 836, 802, 779, 717 cm^{-1};
Anal. Calcd for $C_{23}H_{40}O_6Si$: C, 62.69; H, 9.15. Found: C, 62.86; H, 9.40.

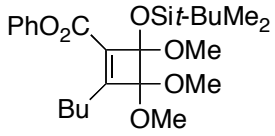
*Synthesis of cyclobutene **8d**:*
According to the general procedure for synthesis of cyclobutene **8a**, alkyne **7d** (118 mg, 0.521 mmol), KSA **2** (167 mg, 0.672 mmol), and Me_3Al (1.03 M in hexane, 0.50 mL, 0.51 mmol) gave, after purification by PTLC (hexane/EtOAc = 8/2), **8d** (182 mg, 73.5%) as a colorless oil.



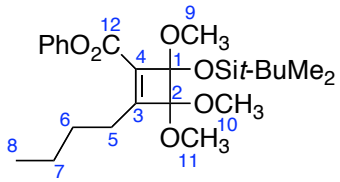
^{1}H NMR (acetone-d_6, δ)
0.16 (s, 3H), 0.19 (s, 3H), 0.92 (s, 9H), 1.56–1.72 (m, 4H), 2.16–2.26 (m, 2H), 2.38–2.46 (m, 2H),

3.455 (s, 3H), 3.459 (s, 3H), 3.53 (s, 3H), 6.68–6.72 (m, 1H), 7.14–7.32 (m, 3H), 7.42–7.48 (m, 2H);
^{13}C NMR (acetone-d_6, δ)
–3.0, –2.8, 19.0, 22.1, 23.0, 26.3, 26.6, 27.2, 52.3, 52.7, 53.0, 106.9, 109.2, 122.5, 126.8, 130.3, 132.1 132.7, 138.6, 151.3, 159.0, 161.6;
IR (KBr)
3062, 2935, 2857, 1731, 1619, 1592, 1492, 1249, 1191, 1164, 1087, 1018, 998, 952, 890, 836, 802, 779 cm^{-1};
Anal. Calcd for $C_{26}H_{38}O_6Si$: C, 65.79; H, 8.07. Found: C, 65.58; H, 8.32.

*Synthesis of cyclobutene **11a**:*
According to the general procedure for synthesis of cyclobutene **8a**, ynoate **10a** (103 mg, 0.509 mmol), KSA **2** (153 mg, 0.616 mmol), and Me_3Al (1.03 M in hexane, 0.50 mL, 0.51 mmol) gave, after purification by PTLC (hexane/CH_2Cl_2/Et_2O = 70/15/15), **11a** (210 mg, 91.5%) as a colorless oil.



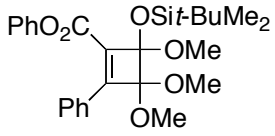
cyclobutene **11a**
^{1}H NMR (acetone-d_6, δ)
0.17 (s, 3H), 0.19 (s, 3H), 0.91 (t, 3H, J = 7.3 Hz), 0.93 (s, 9H), 1.36–1.48 (m, 2H), 1.65–1.76 (m, 2H), 2.68 (t, 2H, J = 7.0 Hz), 3.45 (s, 3H), 3.47 (s, 3H), 3.50 (s, 3H), 7.15–7.32 (m, 3H), 7.40–7.50 (m, 2H);
^{13}C NMR (acetone-d_6, δ)
–3.2, –3.0, 13.9, 19.0, 23.4, 26.3, 28.3, 30.2, 51.5, 51.7, 52.9, 106.3, 108.6, 122.4, 126.7, 130.3, 137.3, 151.1, 161.2, 167.5;
IR (neat)
3064, 2958, 2937, 2857, 1737, 1656, 1592, 1492, 1463, 1288, 1247, 1191, 1074, 1024, 937, 838, 781, 736 cm^{-1};
Anal. Calcd for $C_{24}H_{38}O_6Si$: C, 63.97; H, 8.50. Found: C, 63.73; H, 8.35.



Key HMBC correlations

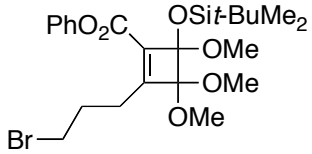
position	δH (ppm)	carbon no. (ppm)
5	2.68	C-2 (108.6)
9	3.50	C-1 (106.3)
10	3.45	C-2 (108.6)
11	3.47	C-2 (108.6)

*Synthesis of cyclobutene **11b**:*
According to the general procedure for synthesis of cyclobutene **8a**, ynoate **10b** (1.09 g, 4.90 mmol), KSA **2** (1.48 g, 5.96 mmol), and Me_3Al (1.05 M in hexane, 4.6 mL, 4.8 mmol) gave, after purification by silica-gel flash column chromatography (hexane/EtOAc = 9/1), cyclobutene **11b** (1.88 g, 81.4%) as a colorless oil.



cyclobutene **11b**
^{1}H NMR (acetone-d_6, δ)
0.19 (s, 3H), 0.24 (s, 3H), 0.96 (s, 9H), 3.43 (s, 3H), 3.44 (s, 3H), 3.61 (s, 3H), 7.18–7.33 (m, 3H), 7.21–7.51 (m, 5H), 8.04–8.16 (m, 2H);
^{13}C NMR (acetone-d_6, δ)
–2.9, –2.7, 19.0, 26.3, 52.4, 52.6, 53.2, 107.0, 109.4, 122.5, 126.9, 129.1, 130.4, 130.6, 131.6, 132.0, 135.2, 151.1, 158.7, 161.4;
IR (neat)
3068, 2938, 2856, 1731, 1631, 1590, 1490, 1276, 1193, 1087, 1022, 995, 894, 836, 781cm^{-1};
Anal. Calcd for $C_{26}H_{34}O_6Si$: C, 66.35; H, 7.28. Found: C, 66.12; H, 7.47.

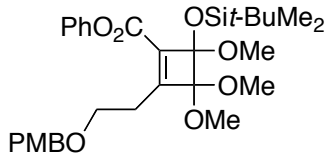
*Synthesis of cyclobutene **11c**:*
According to the general procedure for synthesis of cyclobutene **8a**, ynoate **10c** (127 mg, 0.475 mmol), KSA **2** (151 mg, 0.608 mmol), and Me_3Al (1.03 M in hexane, 0.50 mL, 0.52 mmol) gave, after purification by PTLC (hexane/EtOAc = 7/3), cyclobutene **11c** (209 mg, 85.3%) as a colorless oil.



cyclobutene **11c**
^{1}H NMR (acetone-d_6, δ)
0.16 (s, 3H), 0.19 (s, 3H), 0.92 (s, 9H), 2.18–2.30 (m, 2H), 2.80–2.92 (m, 2H), 3.37 (t, 2H, J = 7.0 Hz), 3.46 (s, 3H), 3.48 (s, 3H), 3.49 (s, 3H), 7.18–7.24 (m, 2H), 7.26–7.34 (m, 1H), 7.42–7.50 (m, 2H);
^{13}C NMR (acetone-d_6, δ)
–3.2, –3.0, 6.9, 19.0, 26.3, 29.7, 32.0, 51.7, 51.9, 52.9, 106.3, 108.7, 122.5, 126.8, 130.3, 138.5, 151.1, 161.1, 165.2;
IR (neat)
2937, 2903, 2856, 2835,1738, 1657, 1592, 1492, 1472, 1462, 1388, 1359, 1283, 1247, 1192, 1162, 1109, 1078, 1041, 1024, 943, 9142 838, 780, 755, 739 cm^{-1};
Anal. Calcd for $C_{23}H_{35}O_6BrSi$: C, 53.59; H, 6.84. Found: C, 53.35; H, 6.60.

*Synthesis of cyclobutene **11d**:*
According to the general procedure for synthesis of cyclobutene **8a**, ynoate **10d** (100 mg, 0.322

mmol), KSA **2** (101 mg, 0.407 mmol), and Me_3Al (1.03 M in hexane, 0.35 mL, 0.36 mmol) gave, after purification by PTLC (hexane/EtOAc = 8/2), cyclobutene **11d** (156 mg, 86.6%) as a colorless oil.



cyclobutene **11d**

^{1}H NMR (acetone-d_6, δ)

0.15 (s, 3H), 0.18 (s, 3H), 0.92 (s, 9H), 2.95 (td, 1H, J_1 = 7.3, J_2 = 14.6 Hz), 2.98 (td, 1H, J_1 = 7.3, J_2 = 14.6 Hz), 3.44 (s, 3H), 3.46 (s, 3H), 3.48 (s, 3H), 3.76 (s, 3H), 3.76 (t, 2H, J = 7.3 Hz), 4.45 (s, 2H), 6.87 (d, 2H, J = 8.7 Hz), 7.08–7.14 (m, 2H), 7.24–7.30 (m, 3H), 7.40–7.46 (m, 2H);

^{13}C NMR (acetone-d_6, δ)

–3.1, –3.0, 19.0, 26.3, 29.4, 51.6, 51.7, 53.0, 55.4, 67.6, 72.8, 106.4, 108.4, 114.3, 122.5, 126.7, 129.9, 130.2, 131.4. 138.9, 151.1, 160.0, 161.1, 163.7;

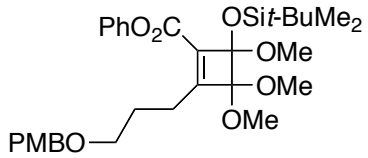
IR (neat)

3066, 2935, 2900, 2857, 1739, 1658, 1612, 1592, 1511, 1492, 1461, 1361, 1284, 1249, 1191, 1083, 1037, 914, 836, 782, 752 cm^{-1};

Anal. Calcd for $C_{30}H_{42}O_8Si$: C, 64.49; H, 7.58. Found: C, 64.30; H, 7.79.

*Synthesis of cyclobutene **11e**:*

According to the general procedure for synthesis of cyclobutene **8a**, alkyne **10e** (151 mg, 0.466 mmol), KSA **2** (147 mg, 0.592 mmol), and Me_3Al (1.03 M in hexane, 0.50 mL, 0.52 mmol) gave, after purification by PTLC (hexane/EtOAc = 8/2), cyclobutene **11e** (245 mg, 91.9%) as a colorless oil.



cyclobutene **11e**

^{1}H NMR (acetone-d_6, δ)

0.16 (s, 3H), 0.18 (s, 3H), 0.92 (s, 9H), 1.94–2.04 (m, 2H), 2.75–2.82 (m, 2H), 3.44 (s, 3H), 3.46 (s, 3H), 3.48 (s, 3H), 3.51 (t, 2H, J = 6.4 Hz), 3.76 (s, 3H), 4.40 (s, 2H), 6.84 (d, 2H, J = 8.7 Hz), 7.14–7.18 (m, 2H), 7.22 (d, 2H, J = 8.7 Hz), 7.25–7.30 (m, 1H), 7.40–7.46 (m, 2H);

^{13}C NMR (acetone-d_6, δ)

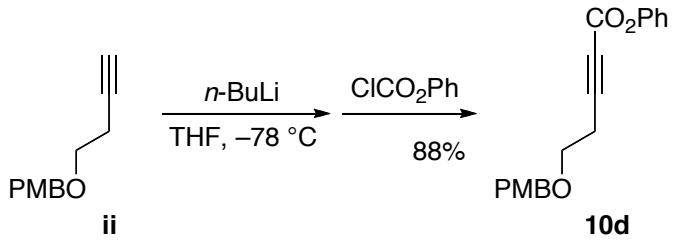
–3.2, –3.0, 19.0, 25.6, 26.3, 28.5, 51.6, 51.8, 52.9, 55.4, 70.0, 72.9, 106.4, 108.6, 114.3, 122.5, 126.7, 129.8, 130.3, 131.8. 137.6, 151.2, 160.0, 161.2, 167.0;

IR (neat)

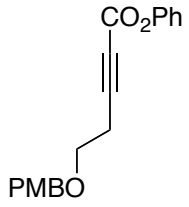
2936, 2855, 1736, 1655, 1612, 1591, 1513, 1492, 1463, 1388, 1360, 1287, 1247, 1190, 1078, 1024, 937, 838, 780, 755 cm^{-1};

Anal. Calcd for $C_{31}H_{44}O_8Si$: C, 65.01; H, 7.74. Found: C, 64.79; H, 7.77.

Scheme 2. Preparation of ynoate **10d**.

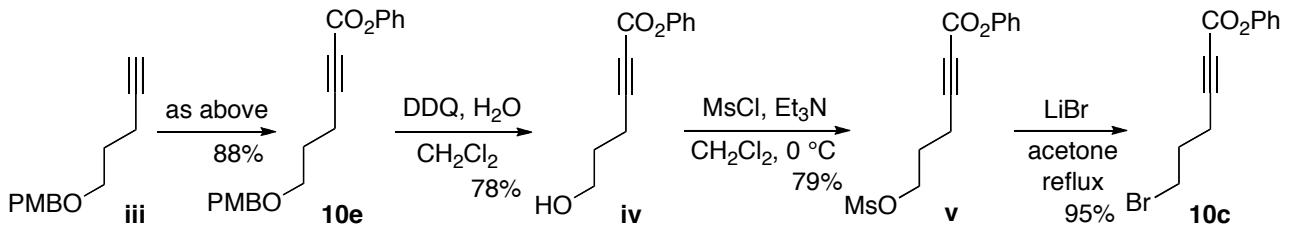


*Synthesis of alkyne **10d**:*
To a mixture of alkyne **ii** (2.45 g, 12.9 mmol) in THF (60 mL) was added *n*-BuLi (1.60 M in hexane, 8.5 mL, 14 mmol) at –78 °C. After 1 h, $ClCO_2Ph$ (2.00 g, 12.8 mmol) in THF (5 mL) was added to the mixture, and the reaction was stirred for further 1 h. The reaction was stopped by adding sat. NH_4Cl. The products were extracted with EtOAc and combined organic extracts were washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by silica-gel flash column chromatography (hexane/EtOAc = 9/1) to give ynoate **10d** (3.50 g, 87.6%) as a colorless oil.



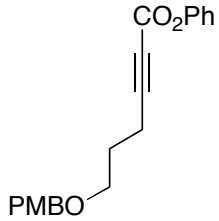
alkyne **10d**
^{1}H NMR ($CDCl_3$, δ)
2.69 (t, 2H, J = 7.3 Hz), 3.65 (t, 2H, J = 7.3 Hz), 3.80 (s, 3H), 4.51 (s, 2H), 6.86–6.94 (m, 2H), 7.10–7.16 (m, 2H), 7.23–7.34 (m, 3H), 7.35–7.44 (m, 2H);
^{13}C NMR ($CDCl_3$, δ)
20.4, 55.3, 66.5, 72.8, 73.5, 88.8, 113.9, 121.4, 126.3, 129.4, 129.5, 129.7, 150.1, 151.8, 159.4;
IR (neat)
3064, 3041, 3002, 2955, 2935, 2913, 2838, 2865, 2236, 1728, 1612, 1589, 1513, 1491, 1457, 1421, 1362, 1328, 1302, 1234, 1190, 1161, 1097, 1043, 1004, 949, 913, 822, 745 cm^{-1};
Anal. Calcd for $C_{19}H_{18}O_4$: C, 73.53; H, 5.85. Found: C, 73.29; H, 6.15.

Scheme 3. Preparation of ynoate **10c** and **10e**.



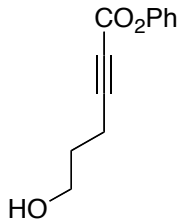
*Synthesis of alkyne **10e**:*
According to the general procedure for synthesis of alkyne **10d**, alkyne **iii** (1.31 g, 6.41 mmol),

$ClCO_2Ph$ (1.12 g, 7.15 mmol), and *n*-BuLi (1.60 M in hexane, 4.1 mL, 6.6 mmol) gave, after purification by silica-gel flash column chromatography (hexane/EtOAc = 9/1), alkyne **10e** (1.83 g, 88.0%) as a colorless oil.



alkyne **10e**
^{1}H NMR ($CDCl_3$, δ)
1.89 (tt, 2H, J_1 = 6.1, J_2 = 7.0 Hz), 2.52 (t, 2H, J = 7.0 Hz), 3.55 (t, 2H, J = 6.1 Hz), 3.79 (s, 3H), 4.45 (s, 2H), 6.86–6.92 (m, 2H), 7.10–7.15 (m, 2H), 7.22–7.30 (m, 3H), 7.36–7.44 (m, 2H);
^{13}C NMR ($CDCl_3$, δ)
15.8, 27.7, 55.3, 67.9, 72.7, 72.9, 91.6, 113.8, 121.4, 126.3, 129.3, 129.5, 130.3, 150.1, 152.0, 159.2;
IR (neat)
3063, 3041, 3001, 2954, 2934, 2858, 2230, 1728, 1612, 1590, 1513, 1490, 1457, 1442, 1363, 1301, 1232, 1190, 1162, 1102, 1063, 1038, 1001, 915, 820, 741 cm^{-1};
Anal. Calcd for $C_{20}H_{20}O_4$: C, 74.06; H, 6.21. Found: C, 74.27; H, 6.32.

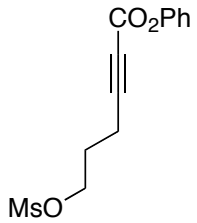
*Synthesis of alkyne **iv**:*
To a solution of ester **10e** (1.12 g, 3.45 mmol) in CH_2Cl_2 (2 mL) and H_2O (1 mL) was added DDQ (812 mg, 3.57 mmol) at 0 °C. After warmed to room temperature, and stirred for further 2 h, the reaction was stopped by adding sat. aq. $NaHCO_3$. The products were extracted with EtOAc and combined organic extracts were washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by silica-gel flash column chromatography (hexane/EtOAc = 7/3) to give alcohol **iv** (547 mg, 77.6%) as a colorless oil.



alcohol **iv**
^{1}H NMR ($CDCl_3$, δ)
1.88 (quint, 2H, J = 7.0 Hz), 2.55 (t, 2H, J = 7.0 Hz), 3.55 (t, 1H, J = 6.0 Hz), 3.78 (dt, 2H, J_1 = 6.0, J_2 = 7.0 Hz), 7.10–7.17 (m, 2H), 7.23–7.28 (m, 1H), 7.36–7.43 (m, 2H);
^{13}C NMR ($CDCl_3$, δ)
15.2, 30.0, 60.8, 72.8, 91.6, 121.3, 126.3, 129.5, 149.9, 152.0;
IR (neat)
3495, 2954, 2881, 2232, 1728, 1590, 1490, 1457, 1425, 1326, 1235, 1190, 1161, 1047, 1001, 915, 838, 742 cm^{-1};
Anal. Calcd for $C_{12}H_{12}O_3$: C, 70.57; H, 5.92. Found: C, 70.42; H, 5.98.

Synthesis of mesylate ***v***

To a mixture of alcohol **iv** (143 mg, 0.700 mmol) and Et_3N (121 mg, 1.20 mmol) in CH_2Cl_2 (3 mL) was added MsCl (91 mg, 0.794 mmol) in CH_2Cl_2 (1 mL) at 0 °C. After 15 min, the reaction was stopped by adding water. The products were extracted with EtOAc and combined organic extracts were washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by silica-gel flash column chromatography (hexane/EtOAc = 5/5) to give mesylate **v** (157 mg, 79.4%) as a colorless oil.



mesylate **v**

^{1}H NMR ($CDCl_3$, δ)

1.89 (quint, 2H, J = 6.3 Hz), 2.60 (t, 2H, J = 6.3 Hz), 3.06 (s, 3H), 4.36 (t, 2H, J = 6.3 Hz), 7.10–7.17 (m, 2H), 7.23–7.30 (m, 1H), 7.37–7.44 (m, 2H);

^{13}C NMR ($CDCl_3$, δ)

15.3, 27.0, 37.4, 67.7, 73.8, 89.3, 121.4, 126.4, 129.6, 150.0, 151.7;

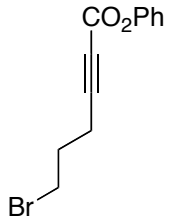
IR (neat)

3029, 2940, 2235, 1727, 1590, 1489, 1355, 1236, 1190, 1175, 972, 929, 832, 748 cm^{-1};

Anal. Calcd for $C_{13}H_{14}O_5S$: C, 55.31; H, 5.00. Found: C, 55.15; H, 5.21.

Synthesis of alkyne ***10c***

To a solution of mesylate **v** (99.2 mg, 0.351 mmol) in acetone (20 mL) was added LiBr (152 mg, 1.75 mmol) at 0 °C. The mixture was heated under reflux conditions for 1 h. The reaction was stopped by adding water, and the products were extracted with EtOAc and combined organic extracts were washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by silica-gel flash column chromatography (hexane/EtOAc = 7/3) to give bromide **10c** (89.1 mg, 94.9%) as a colorless oil.



ynoate **10c**

^{1}H NMR ($CDCl_3$, δ)

2.15 (quint, 2H, J = 6.5 Hz), 2.62 (t, 2H, J = 6.5 Hz), 3.52 (t, 2H, J = 6.5 Hz), 7.10–7.16 (m, 2H), 7.23–7.30 (m, 1H), 7.36–7.44 (m, 2H);

^{13}C NMR ($CDCl_3$, δ)

17.5, 30.2, 31.6, 73.5, 89.8, 121.4, 126.4, 129.6, 150.1, 151.8;

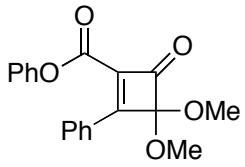
IR (neat)

3064, 2965, 2232, 1728, 1590, 1489, 1456, 1435, 1233, 1190, 1161, 1071, 1038, 1002, 740 cm^{-1};

Anal. Calcd for $C_{12}H_{11}BrO_2$: C, 53.96; H, 4.15. Found: C, 54.06; H, 4.38.

Synthesis of cyclobutenone ***12:***

To a solution of cyclobutene **11b** (456 mg, 0.970 mmol) in CH_2Cl_2 (7.0 mL) and H_2O (0.5 mL) was added $BF_3{\cdot}OEt_2$ (451 mg, 3.19 mmol) in CH_2Cl_2 (3.0 mL) at –78 °C. After the mixture was warmed to –10 °C and stirred for further 0.5 h, the reaction was quenched by adding sat. aq. $NaHCO_3$. The products were extracted with EtOAc (×3), and the combined organic extracts were washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by silica-gel flash column chromatography (hexane/EtOAc = 20/1) to give cyclobutenone **12** (266 mg, 84.7%). Recrystallization from Et_2O gave **12** as colorless prisms.



cyclobutenone **12**

^{1}H NMR (acetone-d_6, δ)

3.55 (s, 6H), 7.29–7.35 (m, 3H), 7.45–7.52 (m, 2H), 7.60–7.66 (m, 2H), 7.70–7.76 (m, 1H), 8.38–8.44 (m, 2H);

^{13}C NMR (acetone-d_6, δ)

54.2, 118.0, 122.5, 127.1, 129.2, 129.9, 130.3, 132.5, 135.7, 138.1, 151.2, 158.9, 183.1, 188.8;

IR (ATR)

3066, 3000, 2942, 2838, 1774, 1735, 1585, 1488, 1450, 1346, 1303, 1253, 1195, 1141, 1103, 1056, 995, 921, 898, 840, 798, 771, 725 cm^{-1};

Anal. Calcd for $C_{19}H_{16}O_5$: C, 70.36; H, 4.97. Found: C, 70.16; H, 4.98.

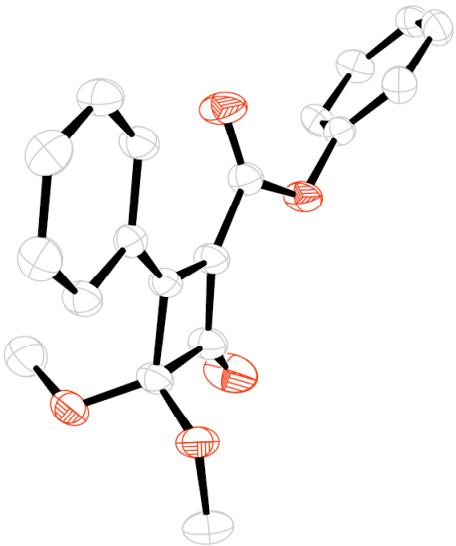
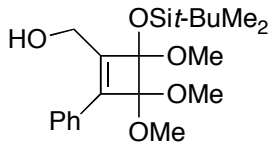


Figure 2. X-ray structure of **12**[7]

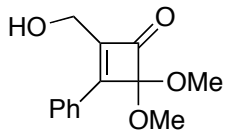
[7] CCDC-709482 (**12**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

*Synthesis of cyclobutene **13:***
To a solution of cyclobutene **11b** (150 mg, 0.319 mmol) in THF (5.0 mL) was added $LiAlH_4$ (20 mg, 0.52 mmol) at 0 °C. After 15 min, the reaction was quenched by adding sat. aq. $NaHCO_3$. The products were extracted with EtOAc (×3), and the combined organic extracts were washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by PTLC (hexane/EtOAc = 7/3) to give cyclobutene **13** (106 mg, 87.4%) as a colorless oil.



cyclobutene **15**
^{1}H NMR (acetone-d_6, δ)
0.17 (s, 6H), 0.92 (s, 9H), 3.35 (s, 3H), 3.47 (s, 6H), 4.05–4.08 (m, 1H), 4.37–4.78 (m, 2H), 7.28–7.40 (m, 3H), 7,72–7.78 (m, 2H);
^{13}C NMR (acetone-d_6, δ)
–2.90, –2.89, 19.0, 26.3, 52.2, 52.7, 55.7, 107.4, 109.8, 128.9, 129.0, 129.4, 133.9, 145.8, 148.4;
IR (neat)
3448, 3056, 2937, 2856, 2834, 1656, 1573, 1494, 1463, 1274, 1253, 1211, 1178, 1139, 1085, 1002, 993, 921, 892, 869, 836, 779 cm^{-1};
Anal. Calcd for $C_{20}H_{32}O_5Si$: C, 63.12; H, 8.48. Found: C, 62.90; H, 8.70.

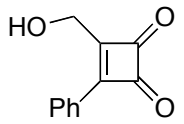
*Synthesis of cyclobutenone **14:***
To a solution of cyclobutene **13** (88.0 mg, 0.231 mmol) in CH_3CN (3.0 mL) was added sat. aq. KF (0.1 mL) and *n*-Bu_4NCl (4.8 mg, 0.0173 mmol) at room temperature. After 24 h, the reaction was stopped by adding water. The products were extracted with EtOAc (×3), and the combined organic extracts were washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by PTLC (hexane/EtOAc = 5/5) to give cyclobutenone **14** (47.2 mg, 87.2%) as a pale yellow oil.



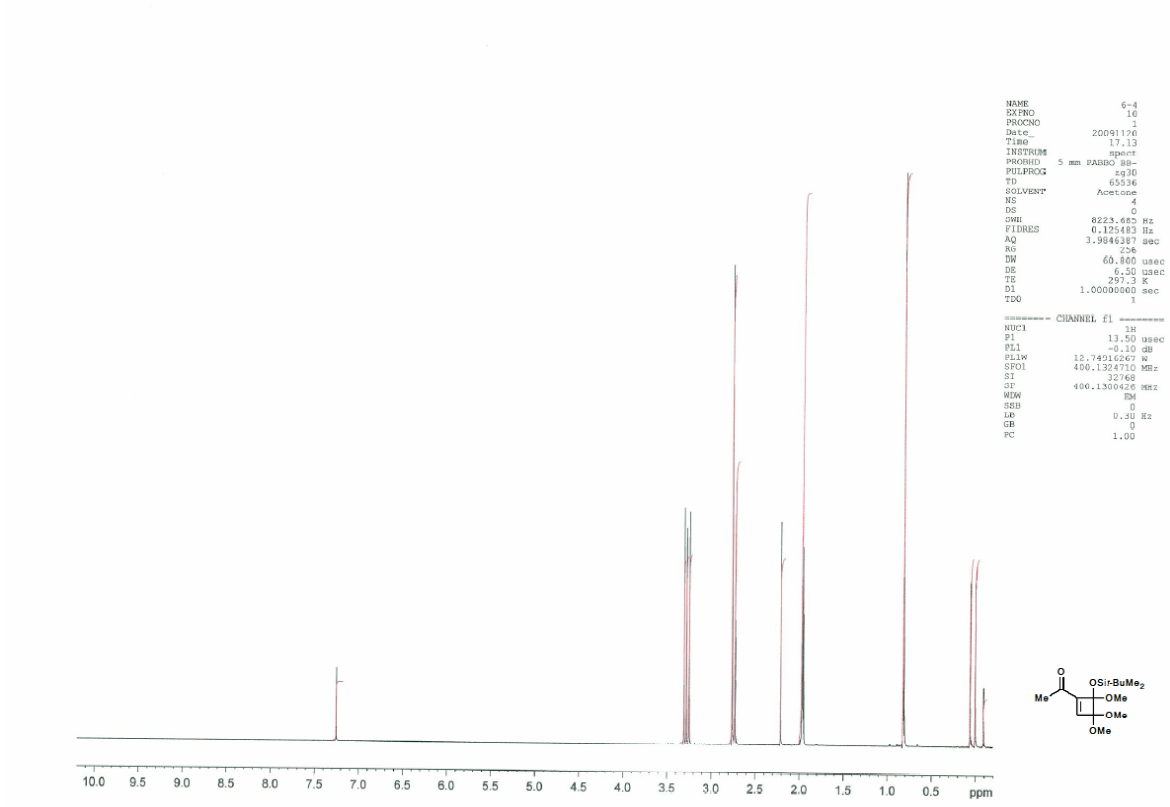
cyclobutenone **14**
^{1}H NMR (acetone-d_6, δ)
3.44 (s, 6H), 4.44–4.52 (m, 3H), 7.50–7.60 (m, 3H), 7.97–8.02 (m, 2H);
^{13}C NMR (acetone-d_6, δ)
53.7, 118.1, 129.6, 130.4, 130.5, 132.6, 152.8, 174.8, 193.9;
IR (neat)
3472, 3064, 2998, 2944, 2837, 1754, 1613, 1573, 1494, 1448, 1346, 1337, 1317, 1302, 1254, 1205, 1171, 1096, 1060, 1026, 1000, 928, 888, 771, 741 cm^{-1};
Anal. Calcd for $C_{13}H_{14}O_4$: C, 66.66; H, 6.02. Found: C, 66.42; H, 5.88.

*Synthesis of cyclobutenedione **15:***
To a solution of cyclobutene **14** (37.3 mg, 0.159 mmol) in CH_2Cl_2 (1.5 mL) and H_2O (0.05 mL) was added $BF_3{\cdot}OEt_2$ (0.05 mL, 0.40 mmol) at 0 °C. After 0.5 h, the reaction was quenched by adding

sat. aq. $NaHCO_3$. The products were extracted with EtOAc (×3), and the combined organic extracts were washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by silica-gel flash column chromatography (hexane/EtOAc = 5/5) to give cyclobutenedione **15** (23.7 mg, 79.1%). Recrystallization from hexane/Et_2O gave **15** as yellow needles. Mp. 101.9–102.6 °C.



cyclobutenedione **15**
^{1}H NMR (acetone-d_6, δ)
4.91 (br s, 1H), 5.10 (s, 2H), 7.58–7.70 (m, 3H), 8.23–8.28 (m, 2H);
^{13}C NMR (acetone-d_6, δ)
57.3, 129.1, 130.0, 130.8, 134.2, 190.5, 196.6, 196.7, 197.8;
IR (ATR)
3472, 3065, 2894, 1780, 1761, 1597, 1581, 1568, 1491, 1448, 1388, 1334, 1312, 1294, 1214, 1184, 1154, 1105, 1085, 1056, 999, 941, 873, 814, 764 cm^{-1};
Anal. Calcd for $C_{11}H_8O_3$: C, 70.21; H, 4.29. Found: C, 69.99; H, 4.03.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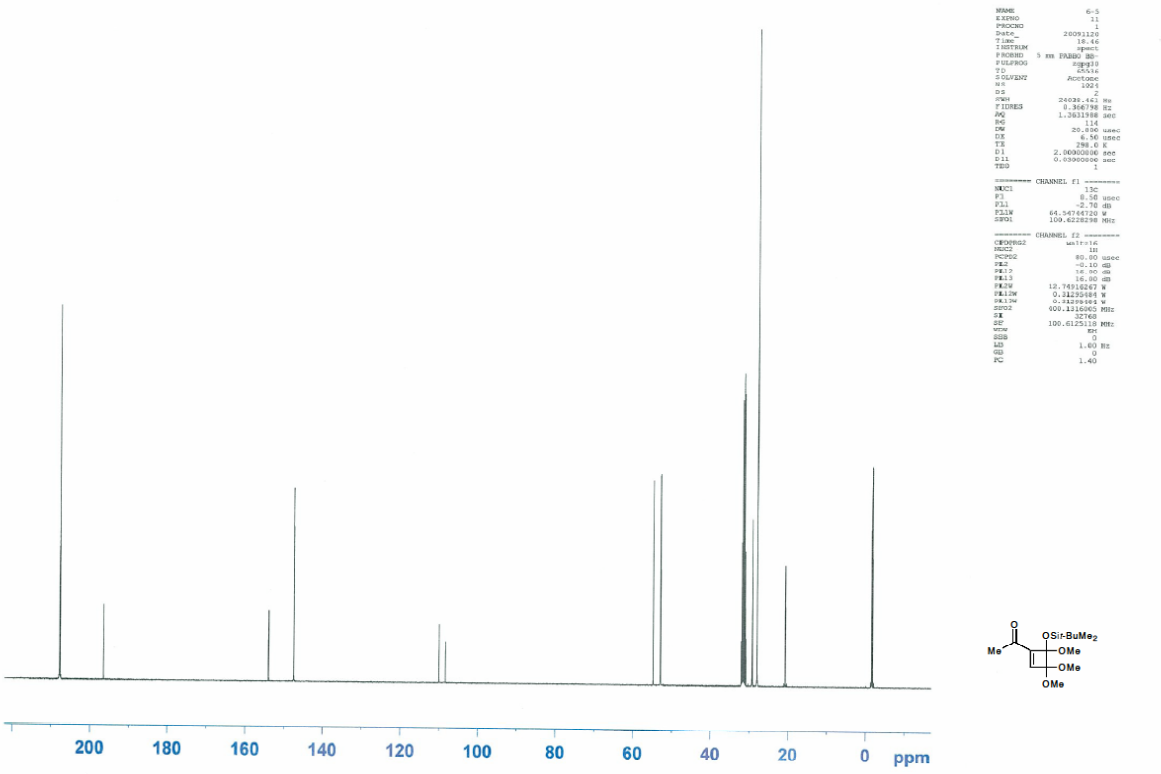
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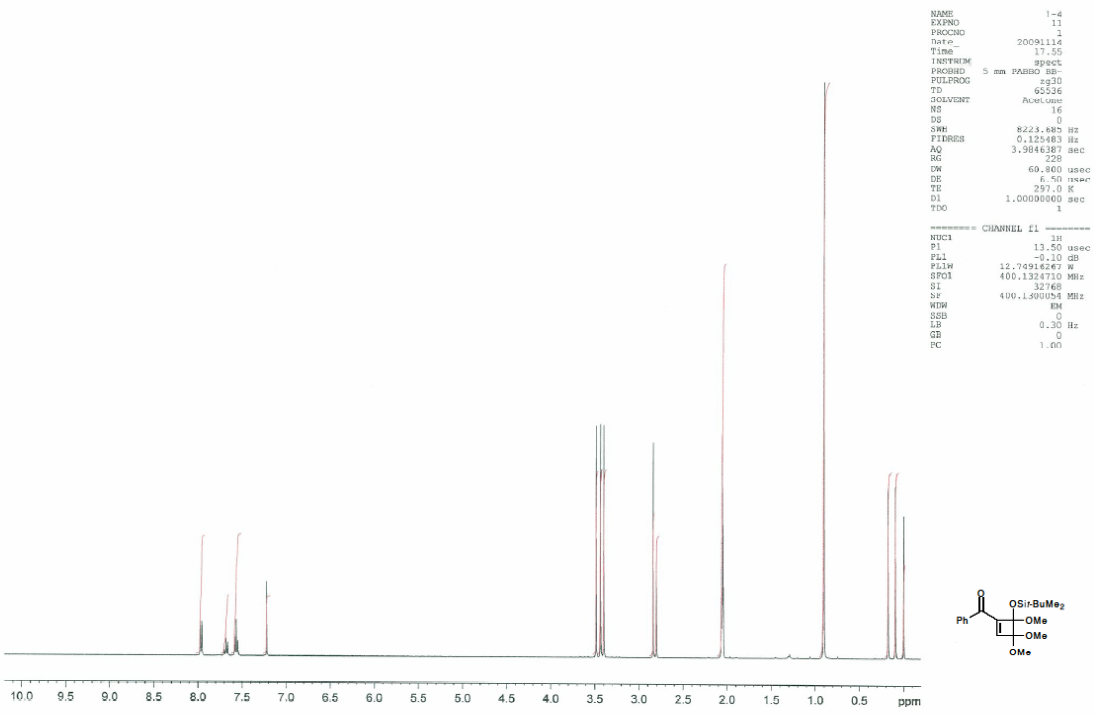
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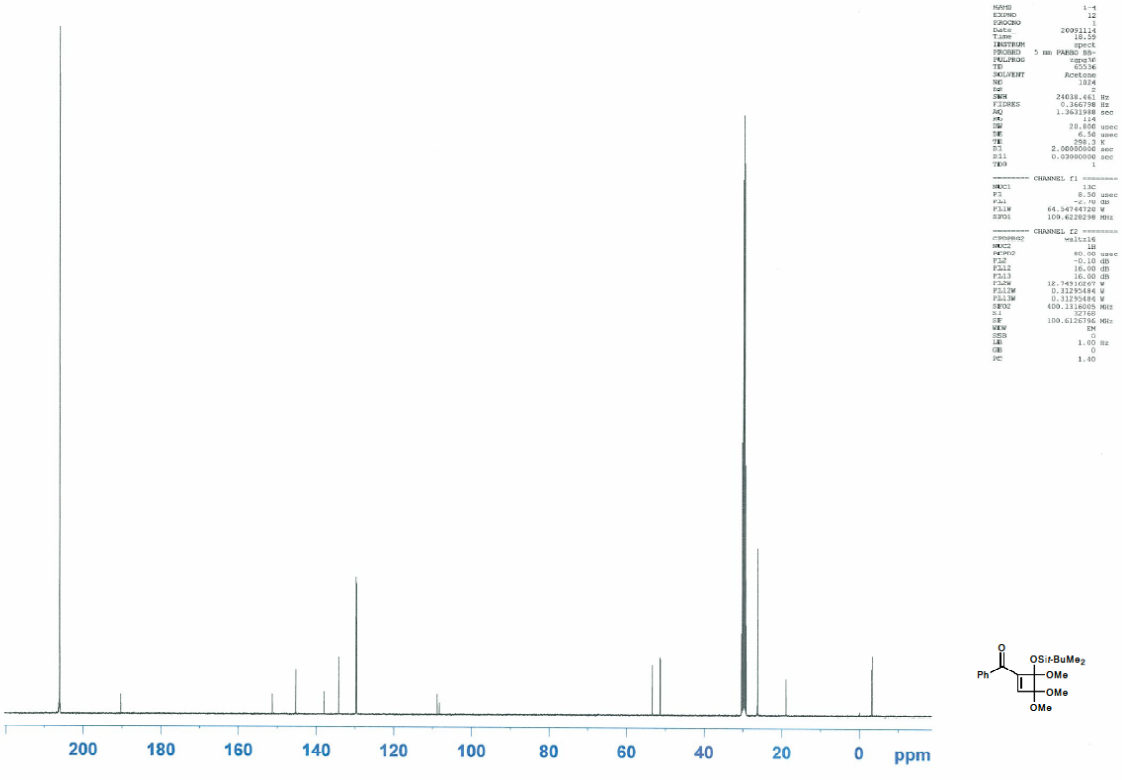
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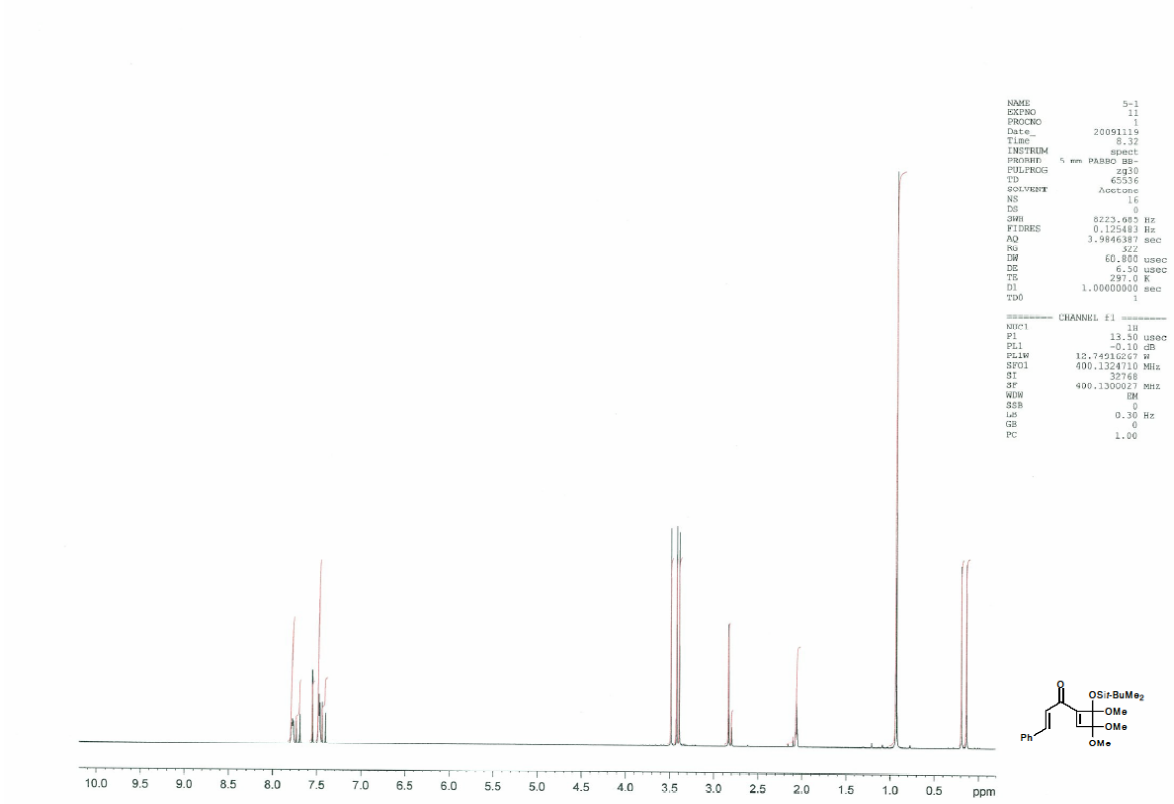
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TE 297.0 K
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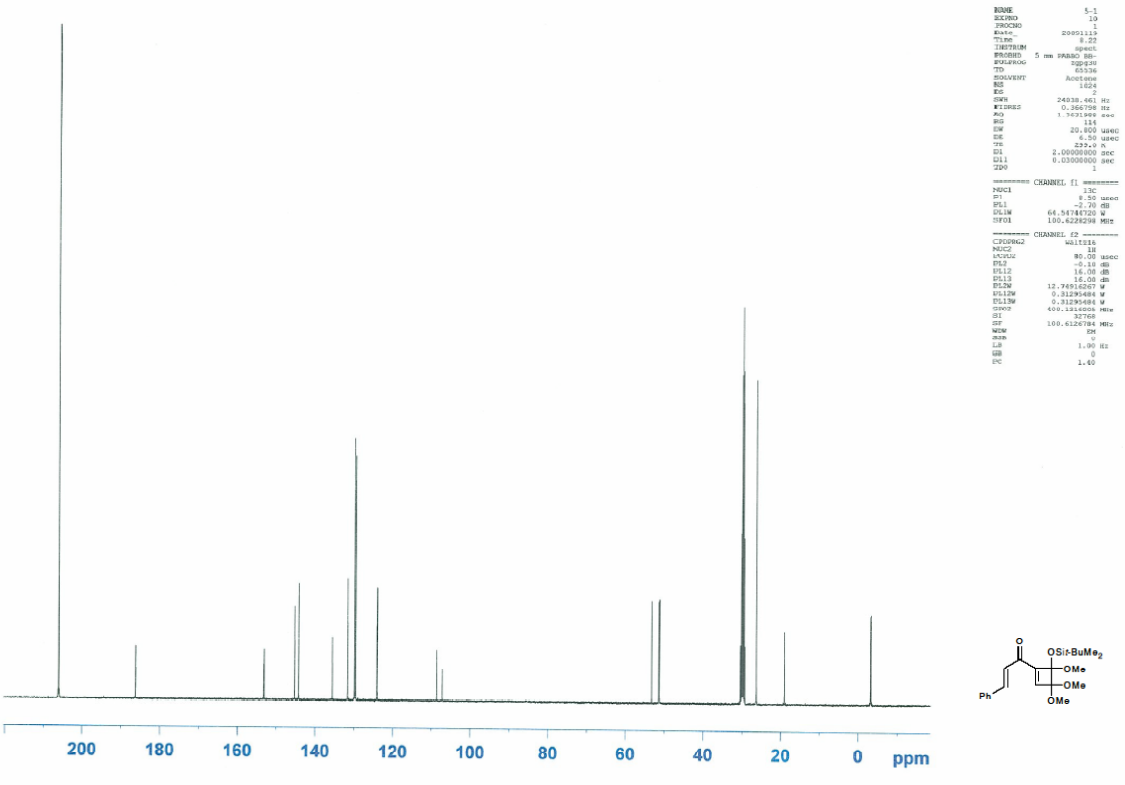
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RG 114
DW 20.800 usec
DE 6.50 usec
TE 299.0 K
D1 2.00000000 sec
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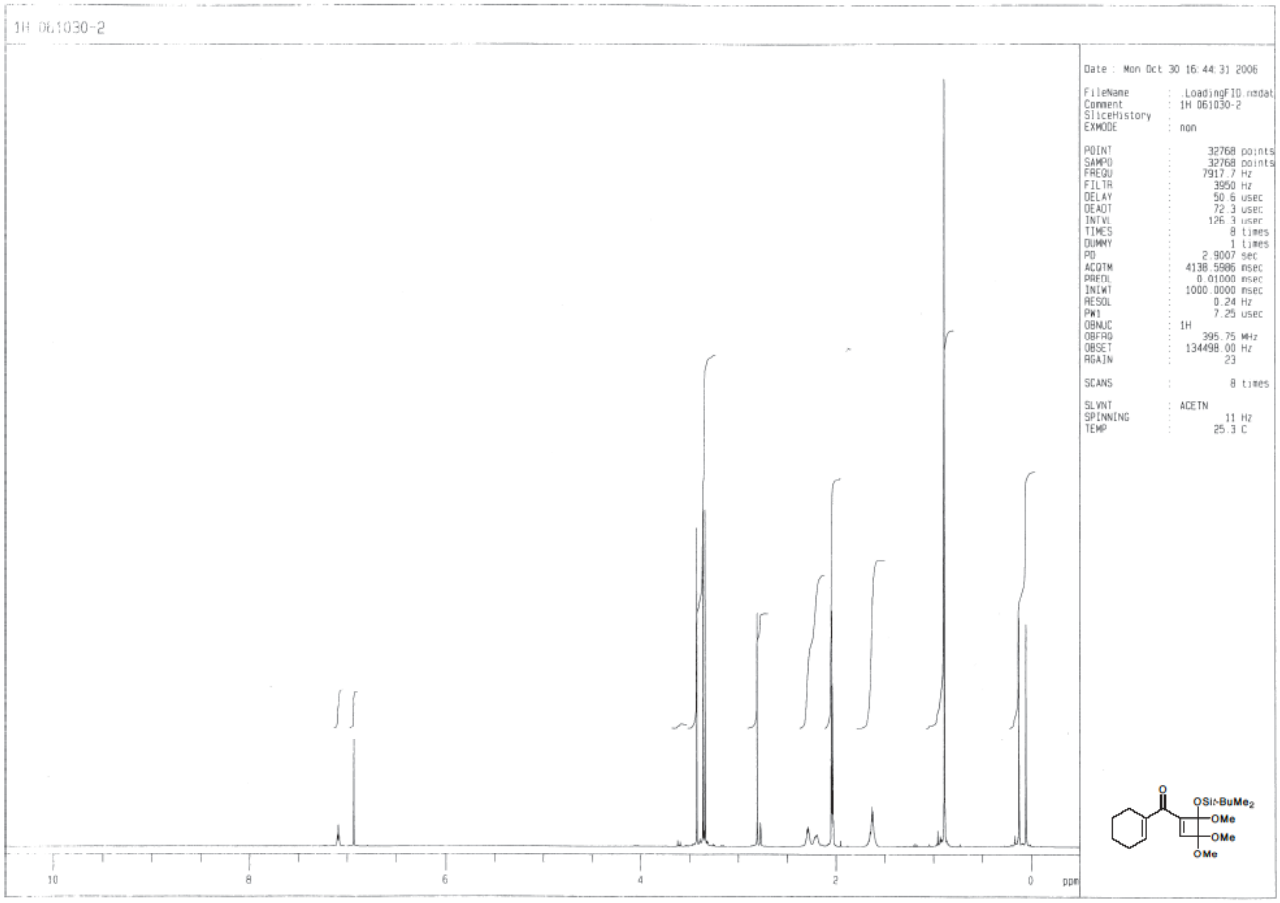
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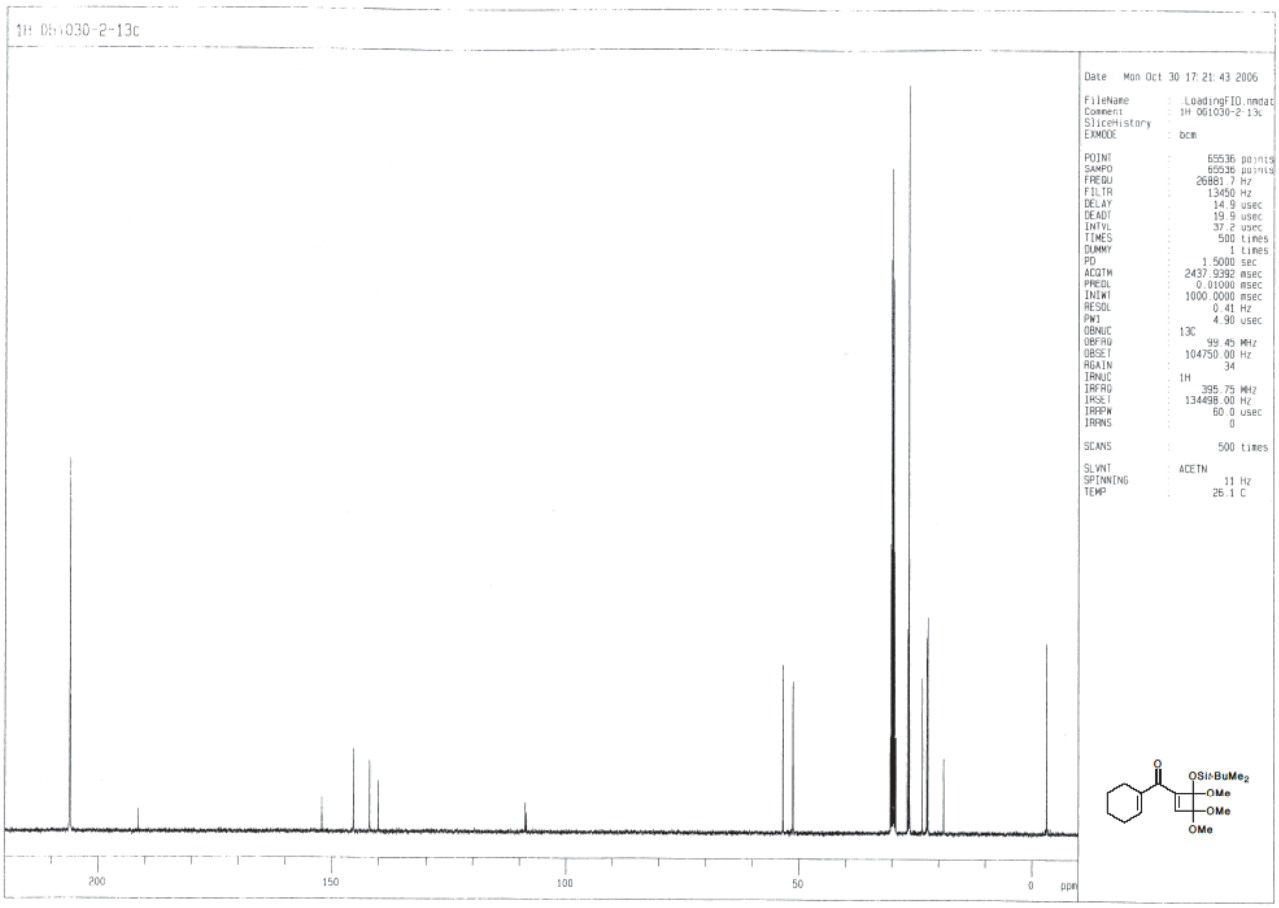
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IRFRQ : 395.75 MHz
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IRPPW : 60.0 usec
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100
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ppm
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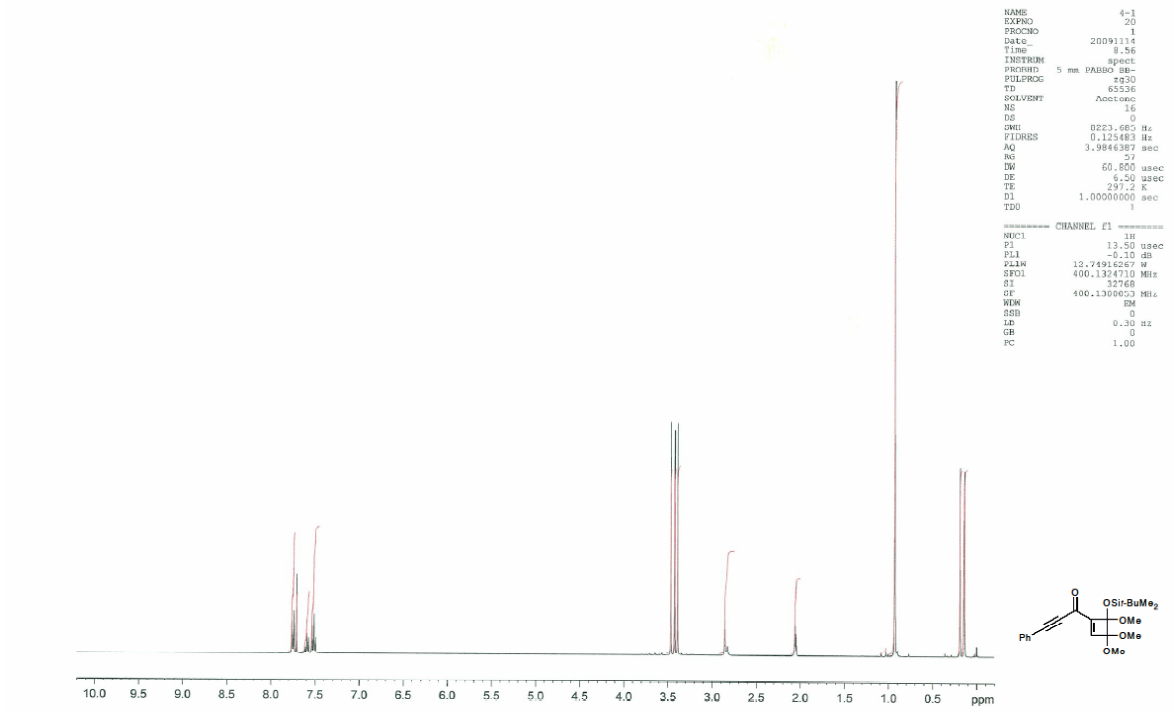
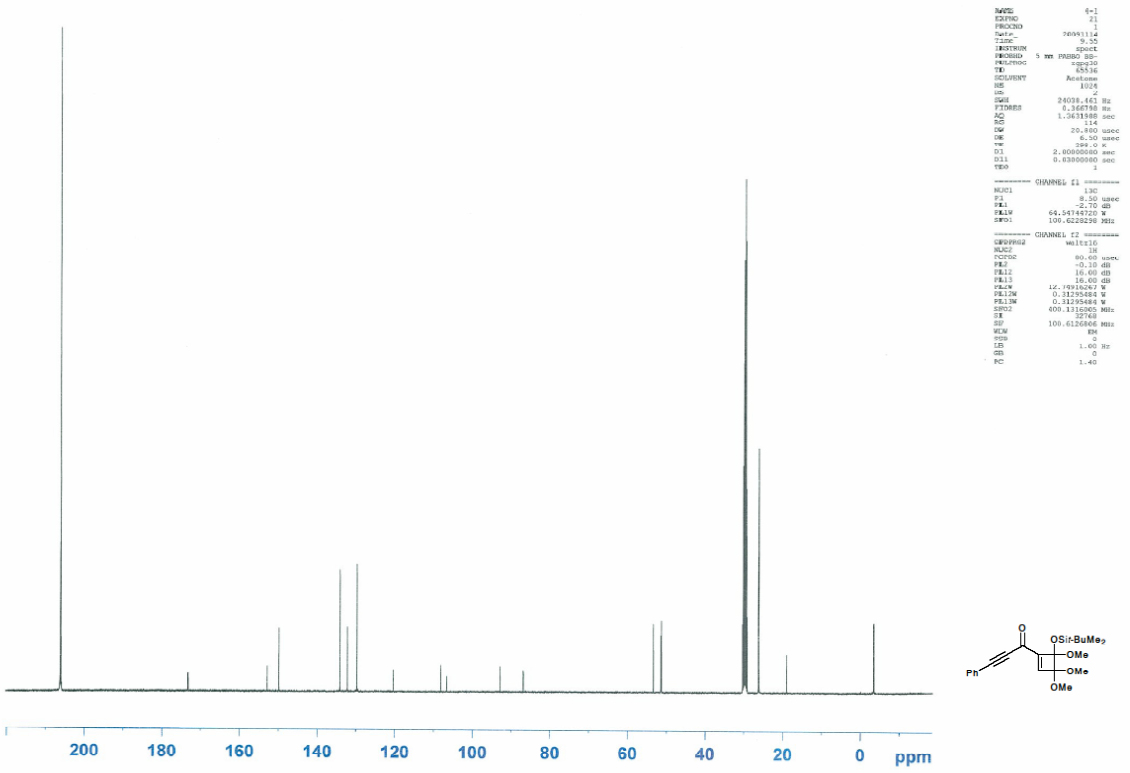
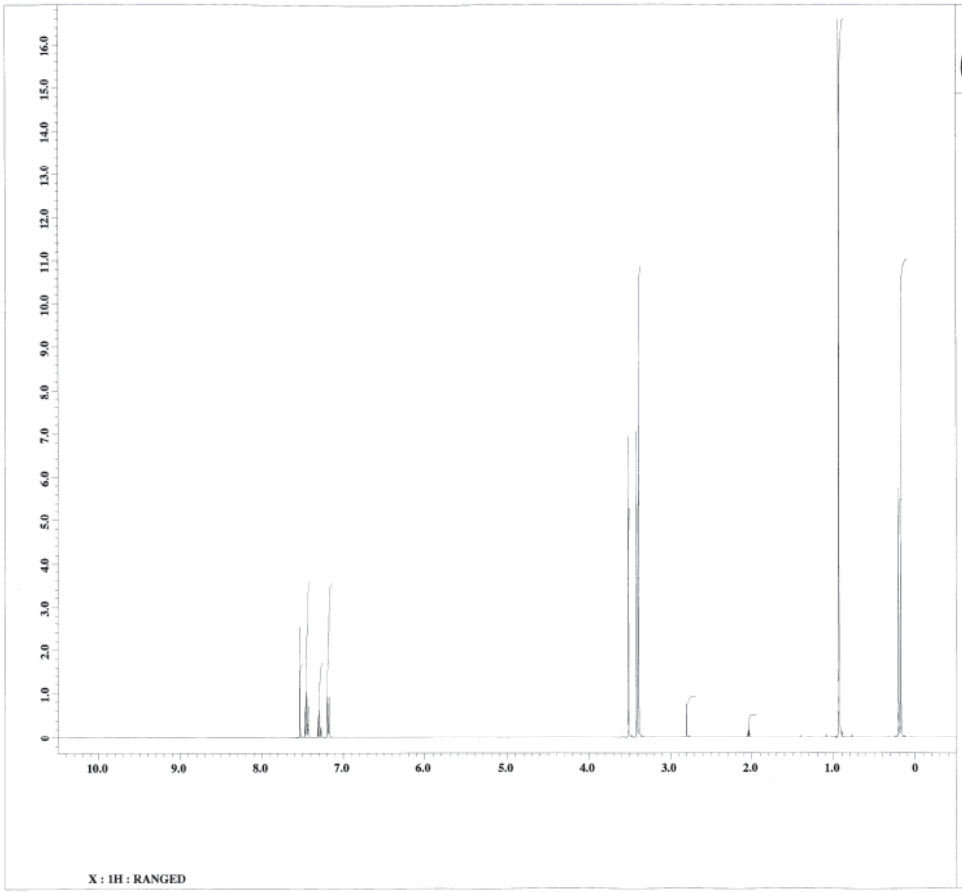
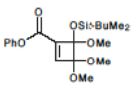
OMe

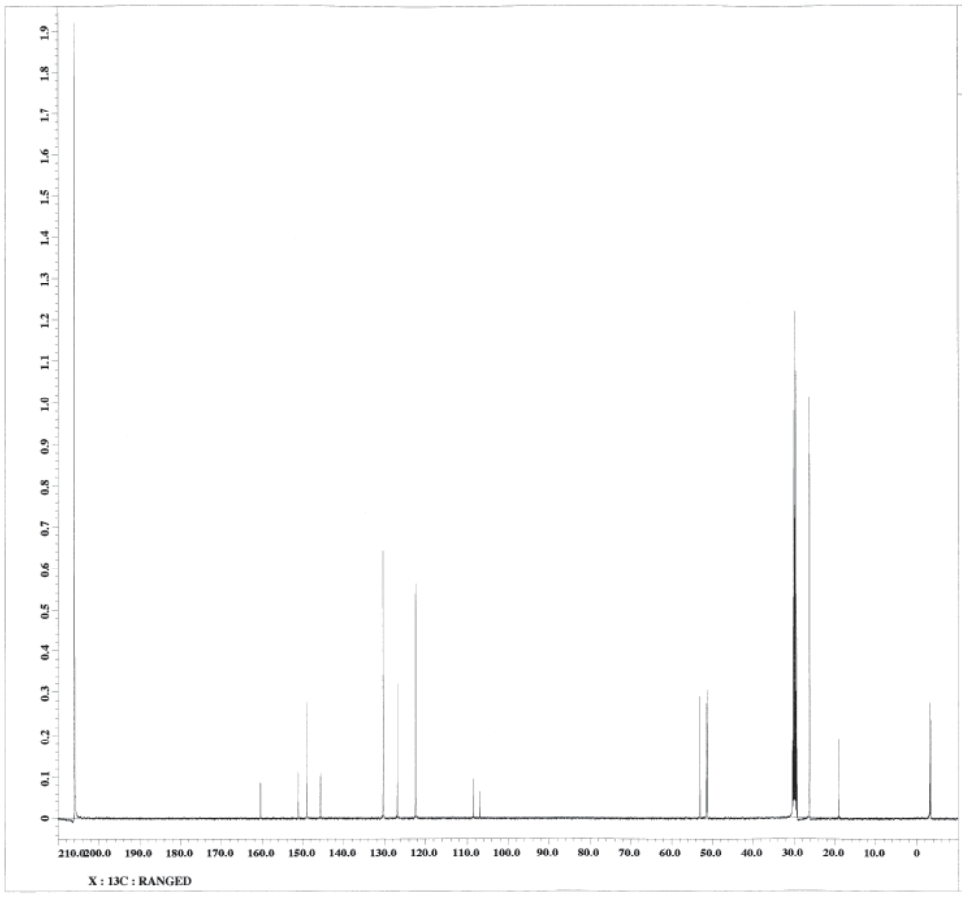

10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
OSit-BuMe2
OMe
OMe
OMe
Ph


200 180 160 140 120 100 80 60 40 20 0 ppm
OSit-BuMe2
OMe
OMe
OMe
Ph

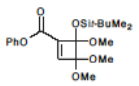


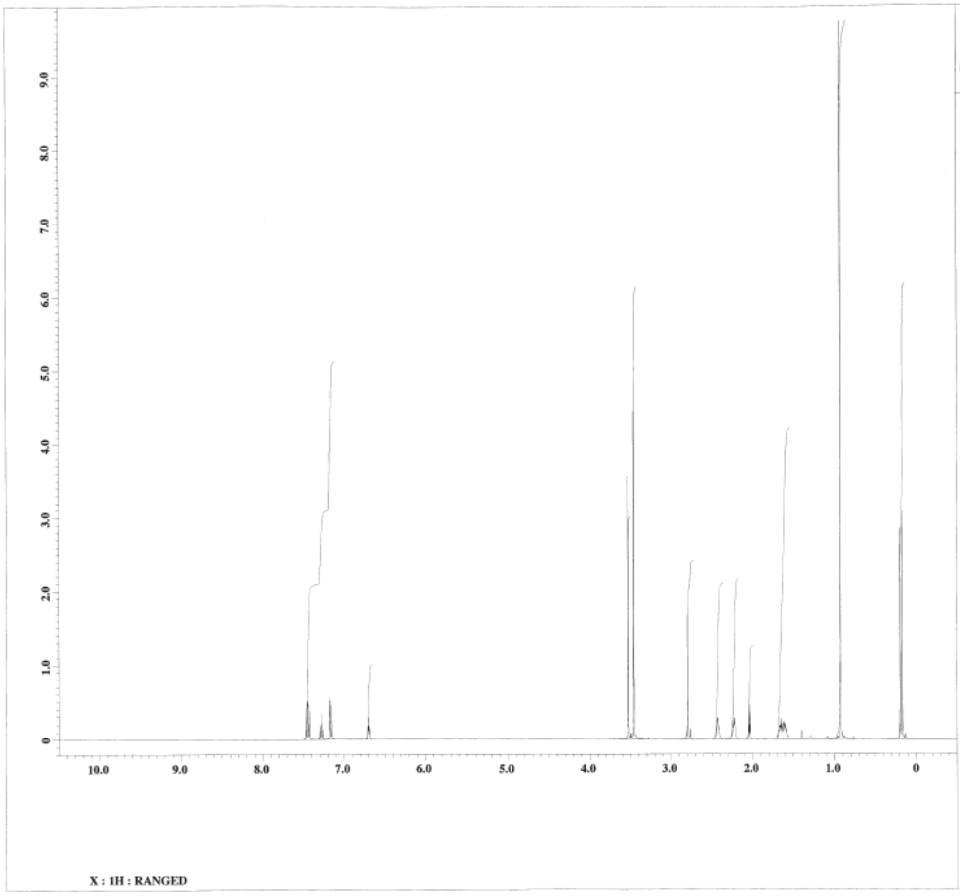

```
Filename          = 090hamura-iwata-Phest
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#543082
Solvent           = ACETONE-D6
Creation_time     = 14-NOV-2009 14:02:19
Revision_time     = 16-NOV-2009 12:03:28
Current_time      = 16-NOV-2009 12:13:26

Comment           = single_pulse
Data_format       = 1D REAL
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 10.5[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 1.4[dB]
X_pulse           = 5.25[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 26
Relaxation_delay  = 5[s]
Repetition_time   = 7.18365952[s]
Temp_get          = 25.2[dC]
```



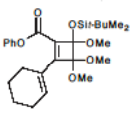


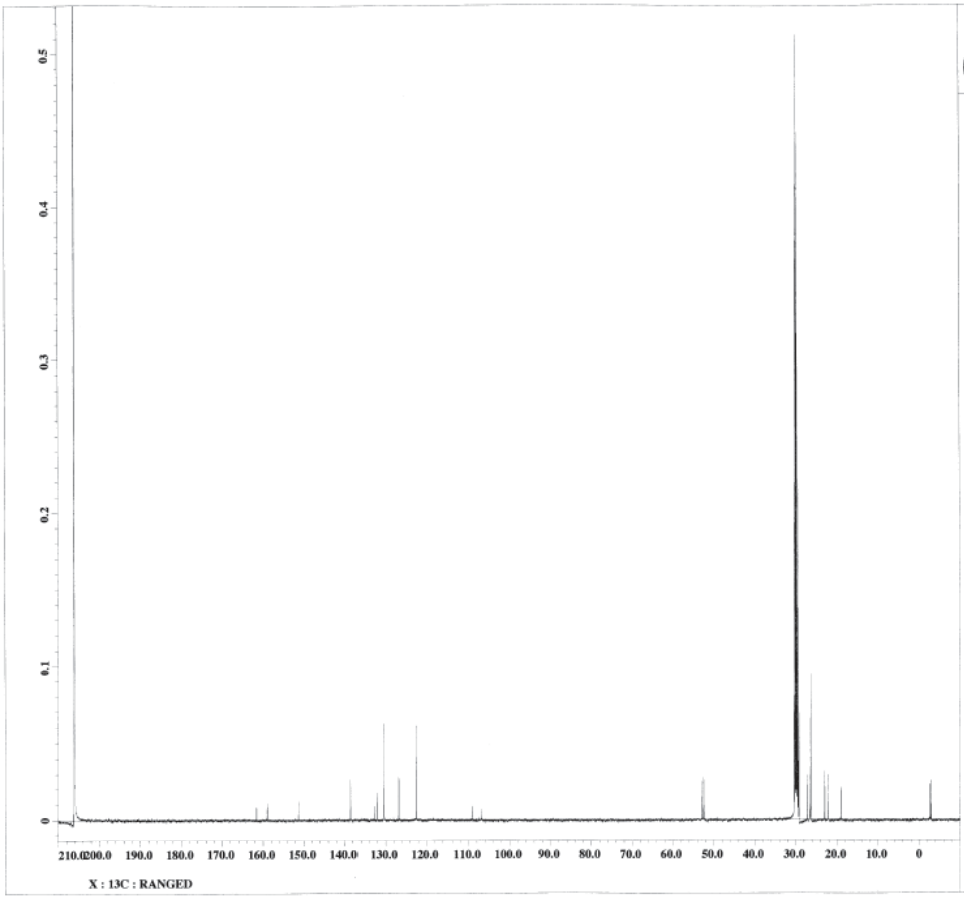

```
Filename          = 090hamura-iwata-Phest
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#518713
Solvent           = ACETONE-D6
Creation_time     = 14-NOV-2009 13:57:34
Revision_time     = 16-NOV-2009 11:53:15
Current_time      = 16-NOV-2009 11:53:49

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 720
Total_scans       = 720

X_90_width        = 9.2[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6.6[dB]
X_pulse           = 3.06666667[us]
Irr_atn_dec       = 22.2[dB]
Irr_atn_noe       = 22.2[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 56
Relaxation_delay  = 2[s]
Repetition_time   = 3.04333312[s]
Temp_get          = 26.3[dC]
```



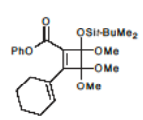


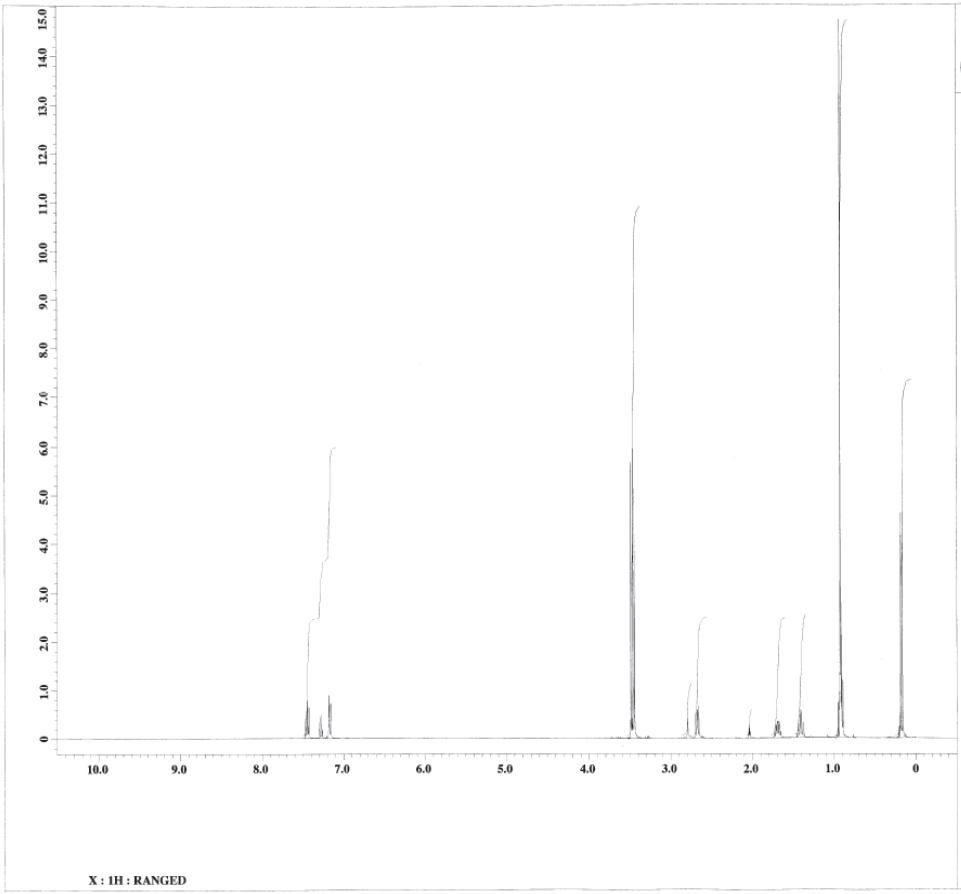

```
Filename          = 090hamura-iwata-cyclo
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#752950
Solvent           = ACETONE-D6
Creation_time     = 12-NOV-2009 19:52:05
Revision_time     = 13-NOV-2009 11:20:43
Current_time      = 13-NOV-2009 11:22:42

Comment           = single_pulse
Data_format       = 1D REAL
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 10.5[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 1.4[dB]
X_pulse           = 5.25[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 32
Relaxation_delay  = 5[s]
Repetition_time   = 7.18365952[s]
Temp_get          = 26.2[dC]
```



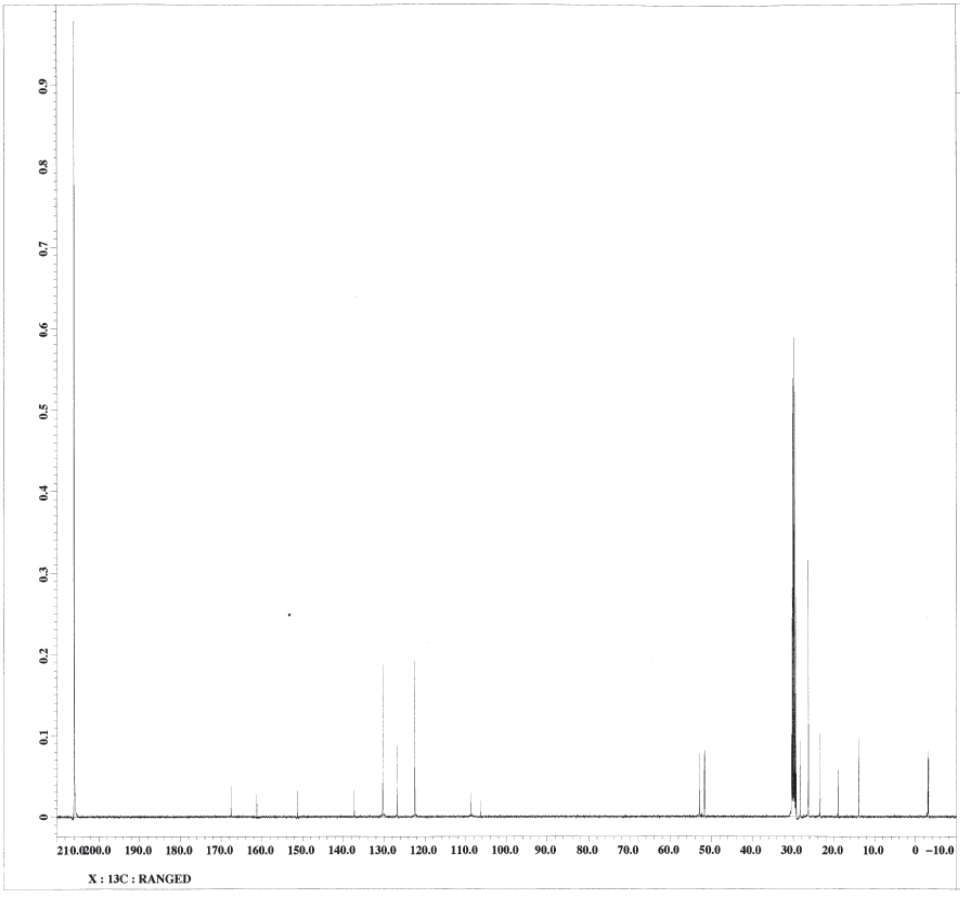



```
Filename          = 090hamura-iwata-cyclo
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#720898
Solvent           = ACETONE-D6
Creation_time     = 12-NOV-2009 19:48:51
Revision_time     = 13-NOV-2009 11:02:52
Current_time      = 13-NOV-2009 11:05:15

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1000
Total_scans       = 1000

X_90_width        = 9.2[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6.6[dB]
X_pulse           = 3.06666667[us]
Irr_atn_dec       = 22.2[dB]
Irr_atn_noe       = 22.2[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 48
Relaxation_delay  = 2[s]
Repetition_time   = 3.04333312[s]
Temp_get          = 26.4[dC]
```

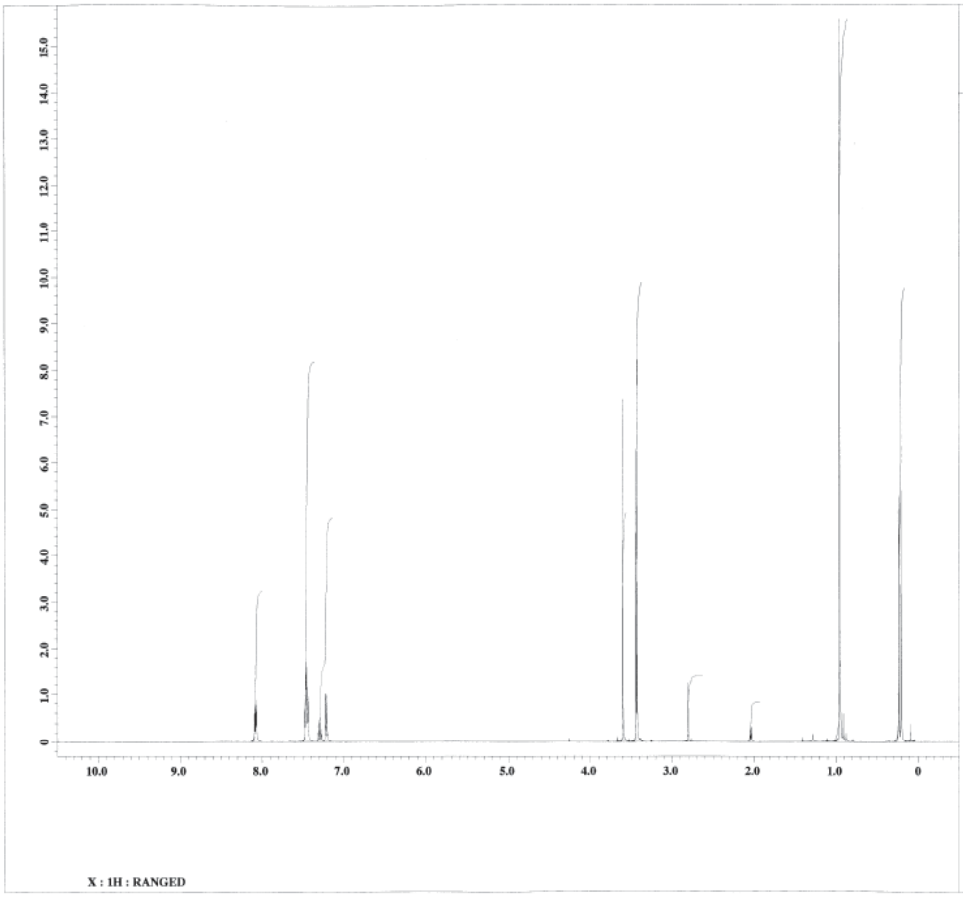




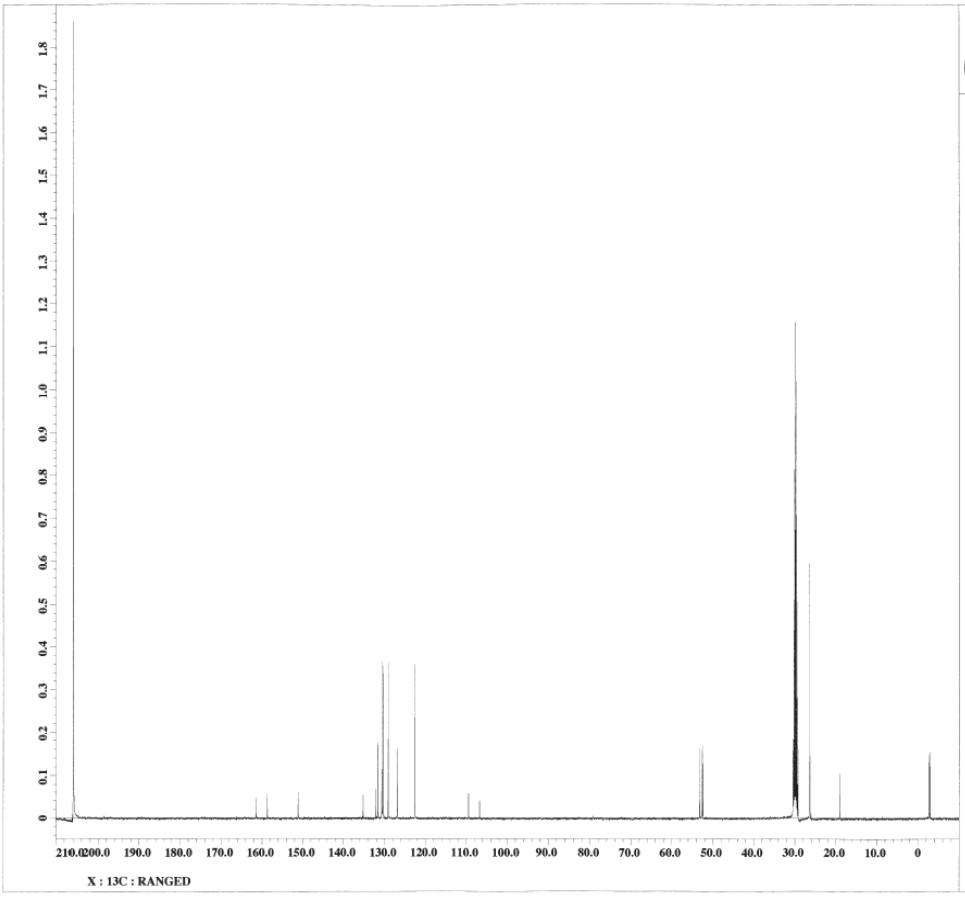

```
Filename          = 090hamura-iwata-BulH-
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#573469
Solvent           = ACETONE-D6
Creation_time     = 11-NOV-2009 14:51:46
Revision_time     = 11-NOV-2009 16:17:24
Current_time      = 11-NOV-2009 16:23:27

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 10.5[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 1.4[dB]
X_pulse           = 5.25[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 28
Relaxation_delay  = 5[s]
Repetition_time   = 7.18365952[s]
Temp_get          = 26.2[dC]
```



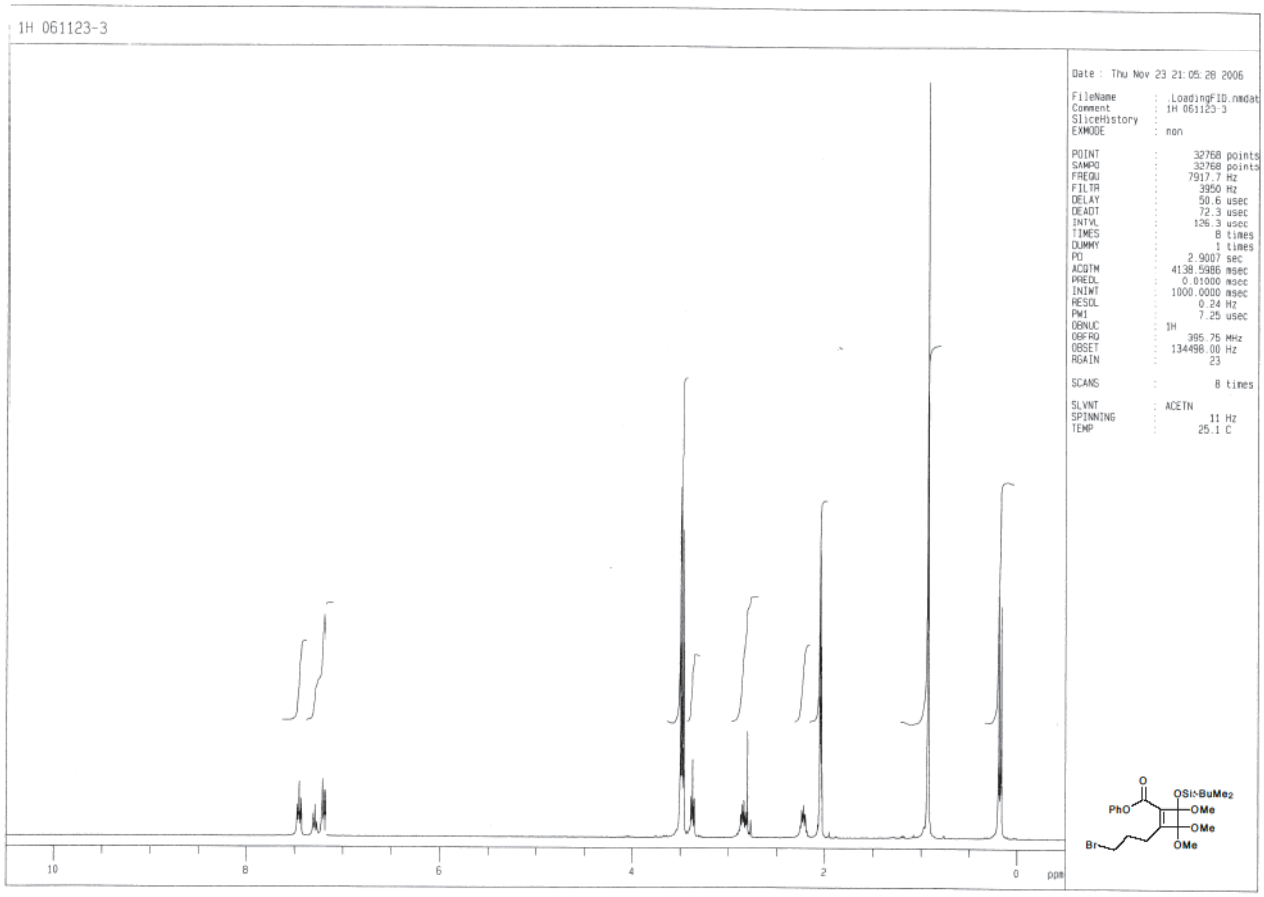

```
Filename          = 090hamura-iwata-Bul3C
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#551614
Solvent           = ACETONE-D6
Creation_time     = 11-NOV-2009 14:46:18
Revision_time     = 11-NOV-2009 16:31:00
Current_time      = 11-NOV-2009 16:31:52

Comment           = single pulse decouple
Data_format       = 1D REAL
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 600
Total_scans       = 600

X_90_width        = 9.2[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6.6[dB]
X_pulse           = 3.06666667[us]
Irr_atn_dec       = 22.2[dB]
Irr_atn_noe       = 22.2[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 50
Relaxation_delay  = 2[s]
Repetition_time   = 3.04333312[s]
Temp_get          = 25.5[dC]
```

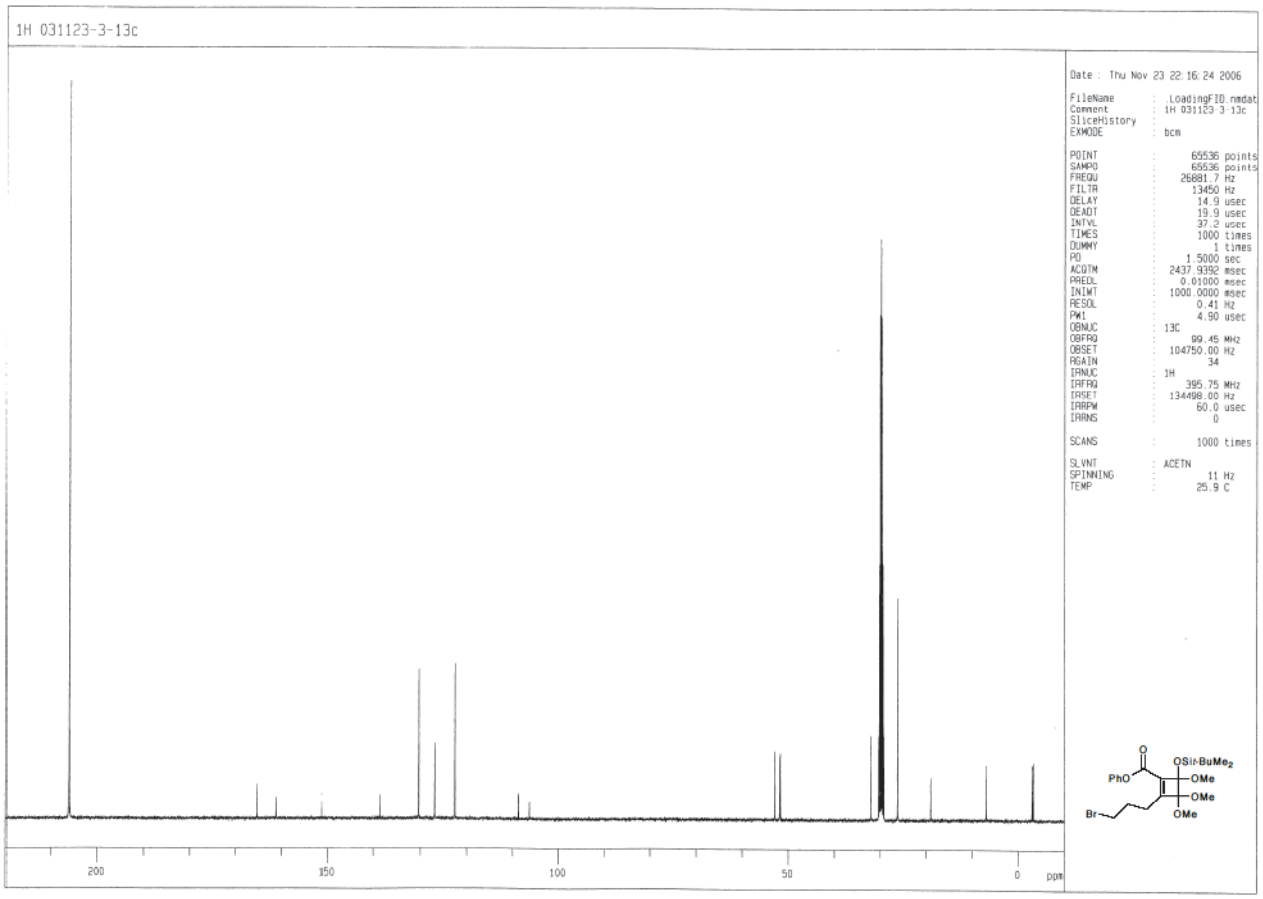


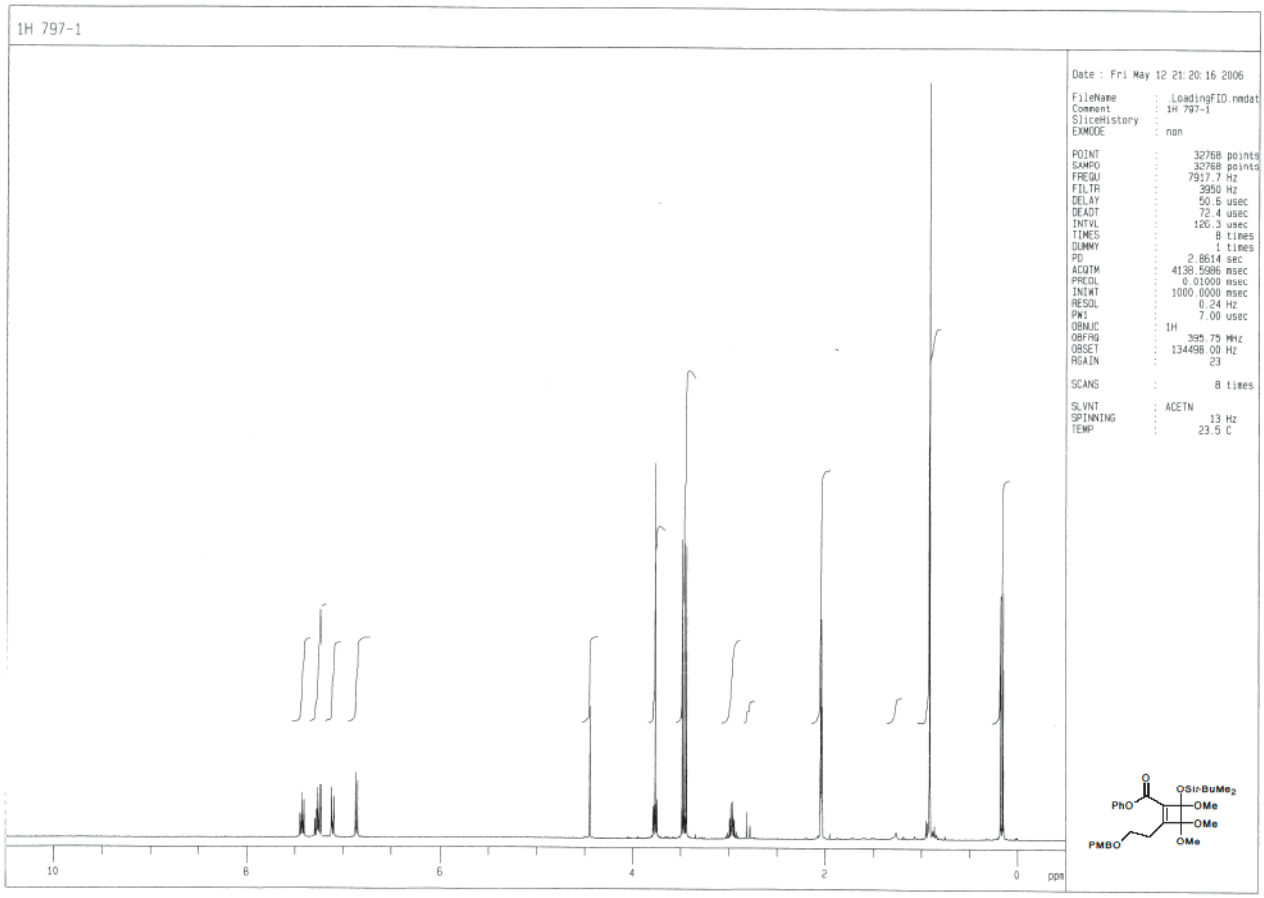

```
Filename          = 090hamura-iwata-PhPhe
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#629483
Solvent           = ACETONE-D6
Creation_time     = 14-NOV-2009 16:25:57
Revision_time     = 16-NOV-2009 12:07:10
Current_time      = 16-NOV-2009 12:09:43

Comment           = single_pulse
Data_format       = 1D REAL
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 10.5[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 1.4[dB]
X_pulse           = 5.25[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 30
Relaxation_delay  = 5[s]
Repetition_time   = 7.18365952[s]
Temp_get          = 26[dC]
```

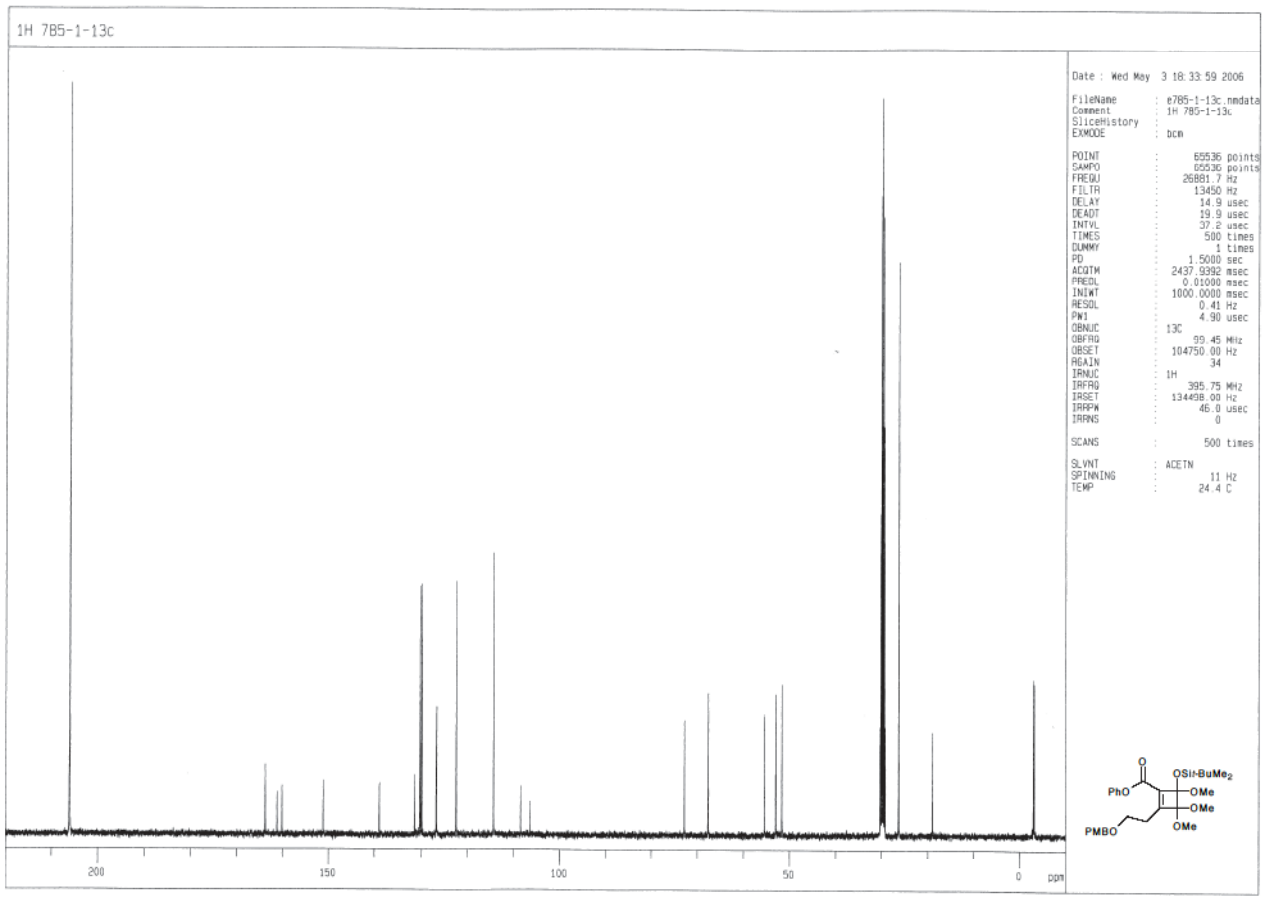


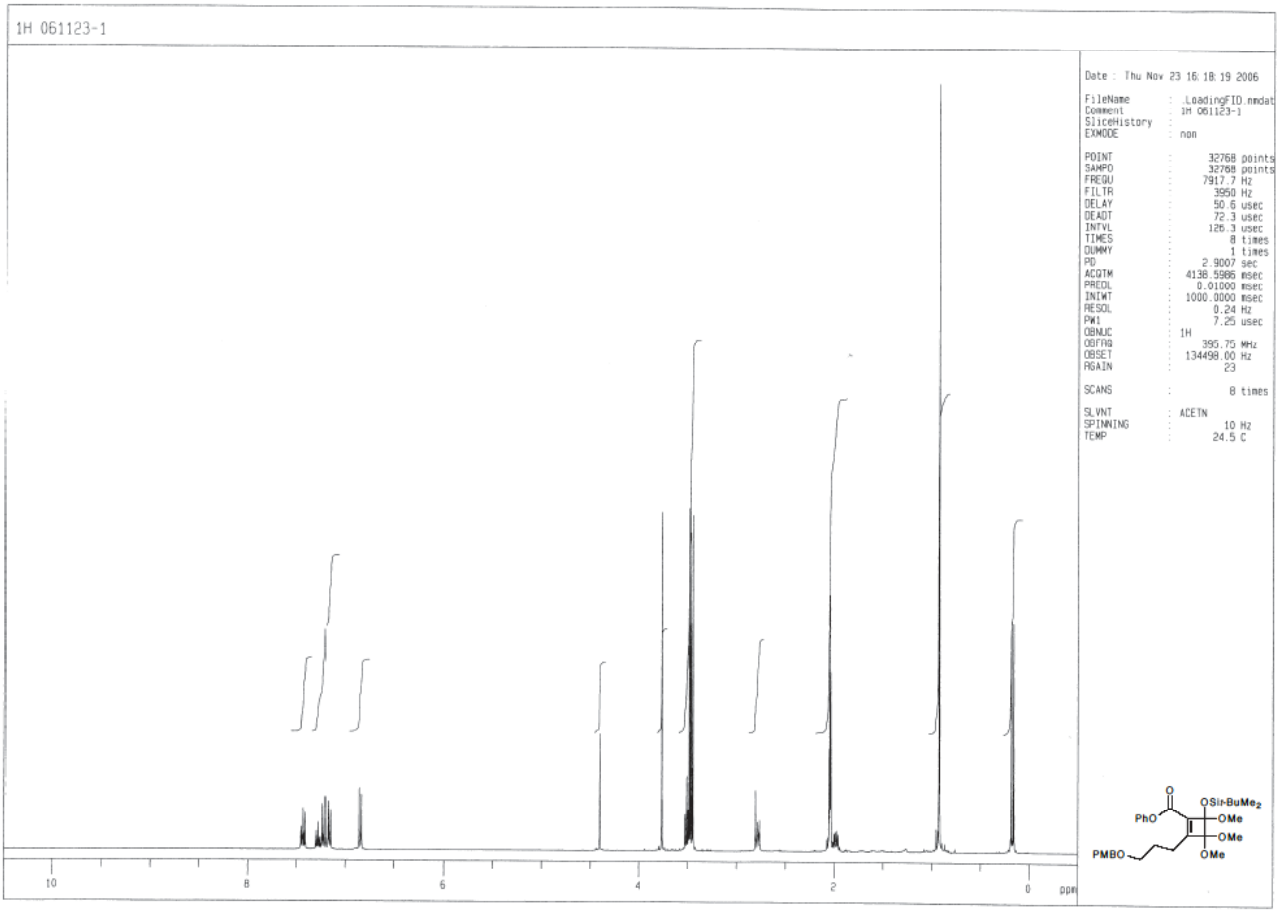

```
Filename          = 090hamura-iwata-PhPhe
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#606134
Solvent           = ACETONE-D6
Creation_time     = 14-NOV-2009 16:21:11
Revision_time     = 16-NOV-2009 11:49:56
Current_time      = 16-NOV-2009 11:50:59

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX400M
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 680
Total_scans       = 680

X_90_width        = 9.2[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6.6[dB]
X_pulse           = 3.06666667[us]
Irr_atn_dec       = 22.2[dB]
Irr_atn_noe       = 22.2[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 56
Relaxation_delay  = 2[s]
Repetition_time   = 3.04333312[s]
Temp_get          = 25.6[dC]
```

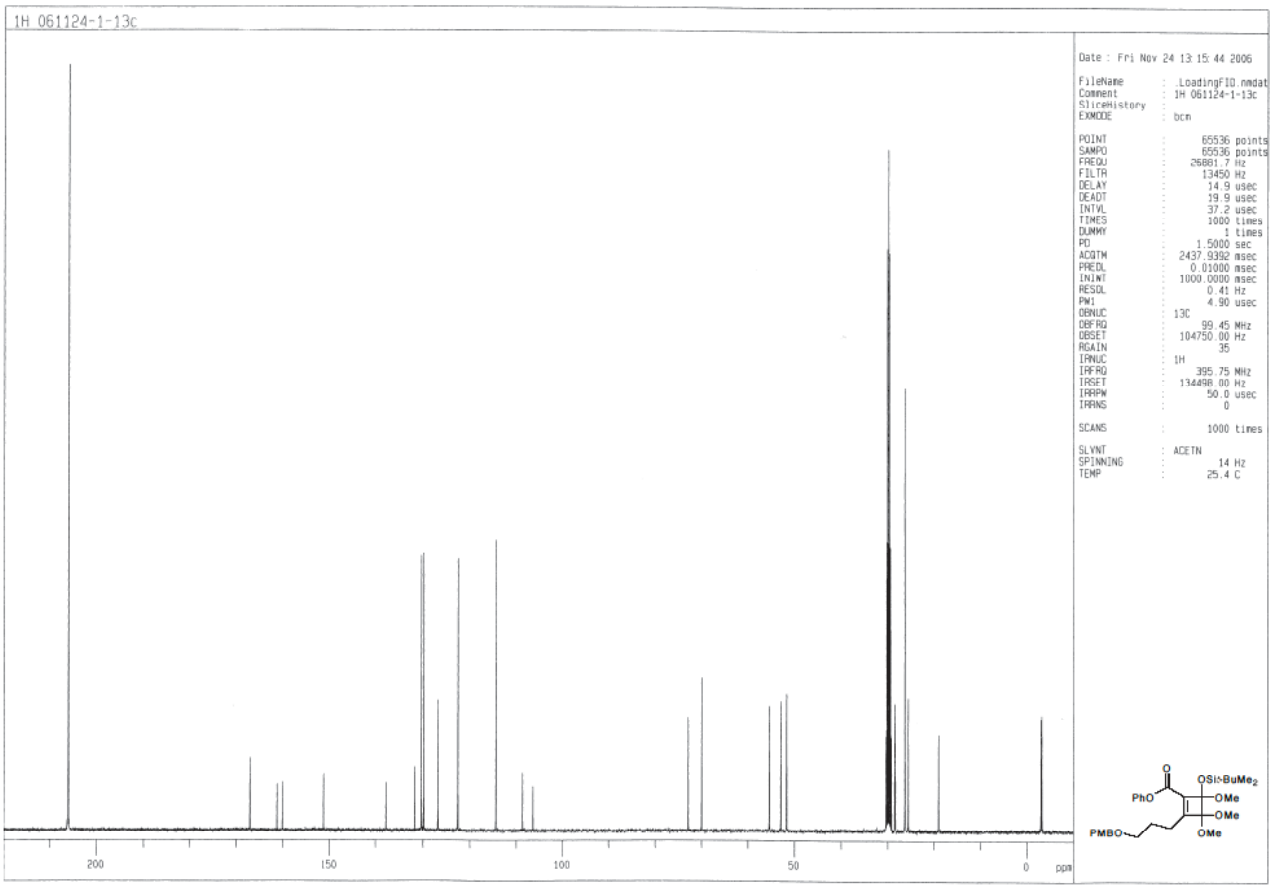

1H 061123-3
Date : Thu Nov 23 21:05:28 2006
FileName : LoadingFID.nmdat
Comment : 1H 061123-3
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7917.7 Hz
FILTR : 3950 Hz
DELAY : 50.6 usec
DEADT : 72.3 usec
INTVL : 126.3 usec
TIMES : 8 times
DUMMY : 1 times
PD : 2.9007 sec
ACQTM : 4138.5986 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.24 Hz
PW1 : 7.25 usec
OBNUC : 1H
OBFRQ : 395.75 MHz
OBSET : 134498.00 Hz
RGAIN : 23
SCANS : 8 times
SLVNT : ACETN
SPINNING : 11 Hz
TEMP : 25.1 C
10
8
6
4
2
0
ppm
PhO
OSi-tBuMe2
OMe
OMe
OMe
Br

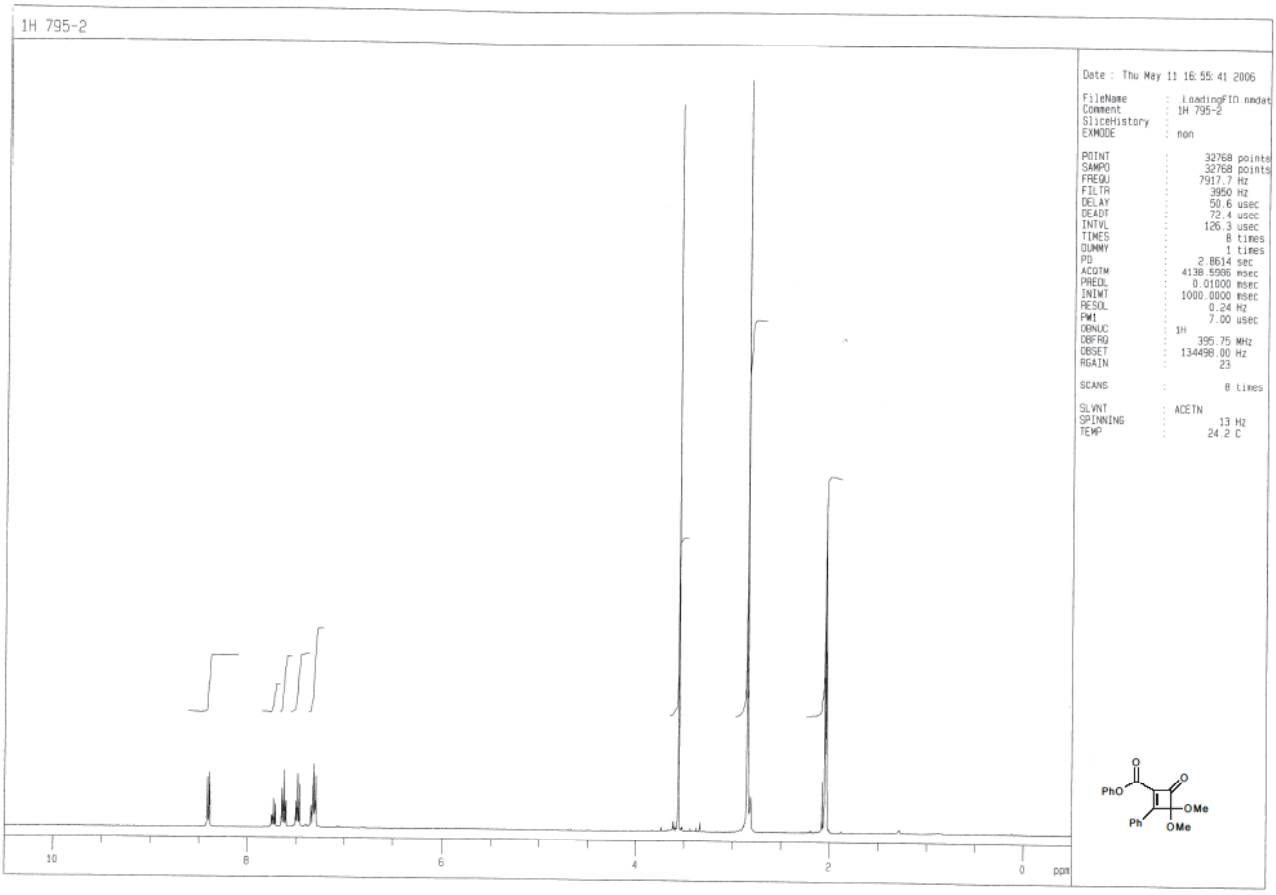

1H 031123-3-13c
Date : Thu Nov 23 22:16:24 2006
FileName : LoadingFID.nmdat
Comment : 1H 031123-3-13c
SliceHistory :
EXMODE : bcm
POINT : 65536 points
SAMPO : 65536 points
FREQU : 26881.7 Hz
FILTR : 13450 Hz
DELAY : 14.9 usec
DEADT : 19.9 usec
INTVL : 37.2 usec
TIMES : 1000 times
DUMMY : 1 times
PD : 1.5000 sec
ACQTM : 2437.9392 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.41 Hz
PW1 : 4.90 usec
OBNUC : 13C
OBFRQ : 99.45 MHz
OBSET : 104750.00 Hz
RGAIN : 34
IRNUC : 1H
IRFRQ : 395.75 MHz
IRSET : 134498.00 Hz
IRRPW : 60.0 usec
IRRNS : 0
SCANS : 1000 times
SLVNT : ACETN
SPINNING : 11 Hz
TEMP : 25.9 C
200
150
100
50
0
ppm
PhO
OSi-tBuMe2
OMe
OMe
OMe
Br

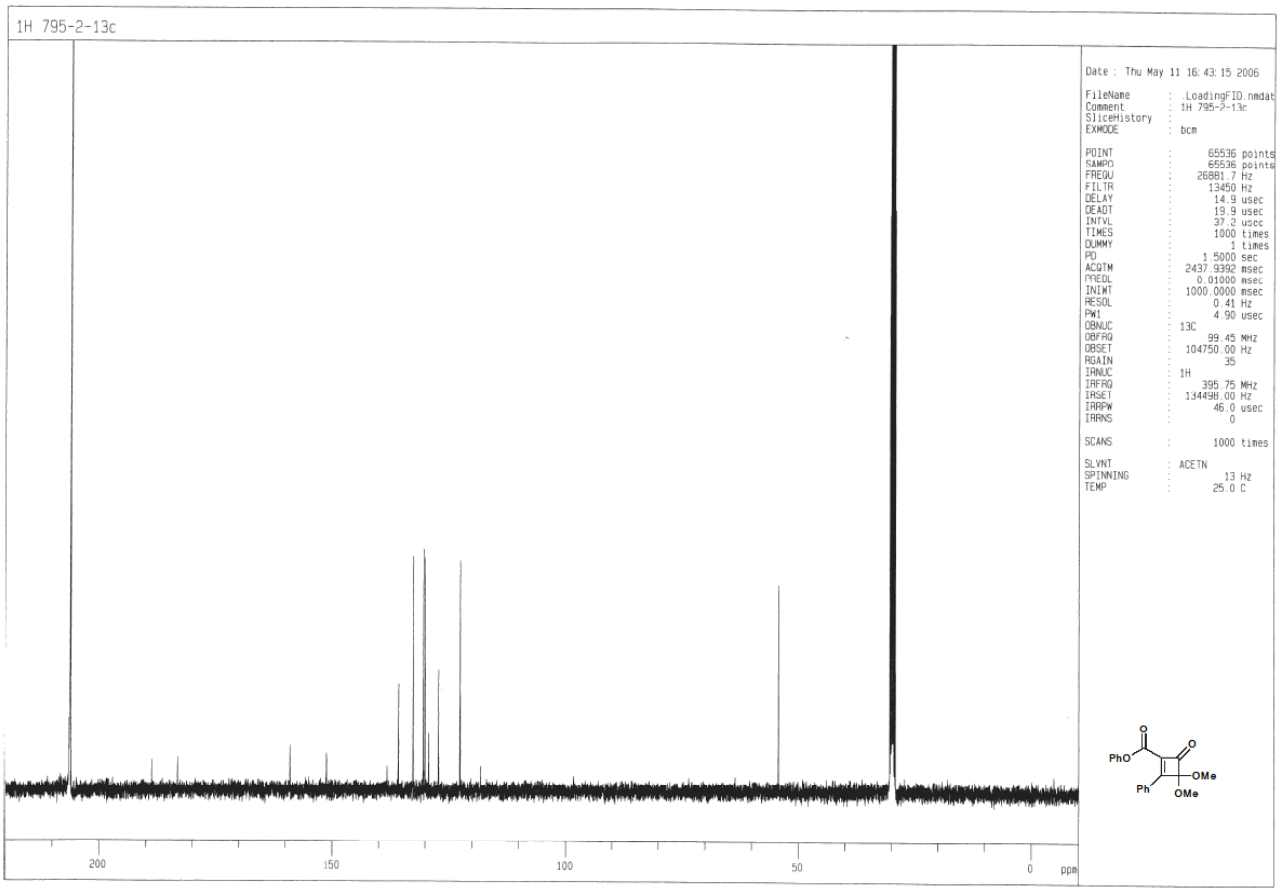

1H 797-1
Date : Fri May 12 21:20:16 2006
FileName : LoadingFID.nmdat
Comment : 1H 797-1
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7917.7 Hz
FILTR : 3950 Hz
DELAY : 50.6 usec
DEADT : 72.4 usec
INTVL : 126.3 usec
TIMES : 8 times
DUMMY : 1 times
PD : 2.8614 sec
ACQTM : 4138.5986 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.24 Hz
PW1 : 7.00 usec
OBNUC : 1H
OBFRQ : 395.75 MHz
OBSET : 134498.00 Hz
RGAIN : 23
SCANS : 8 times
SLVNT : ACETN
SPINNING : 13 Hz
TEMP : 23.5 C
OSi-BuMe2
PhO
OMe
OMe
PMBO
OMe
10
8
6
4
2
0
ppm

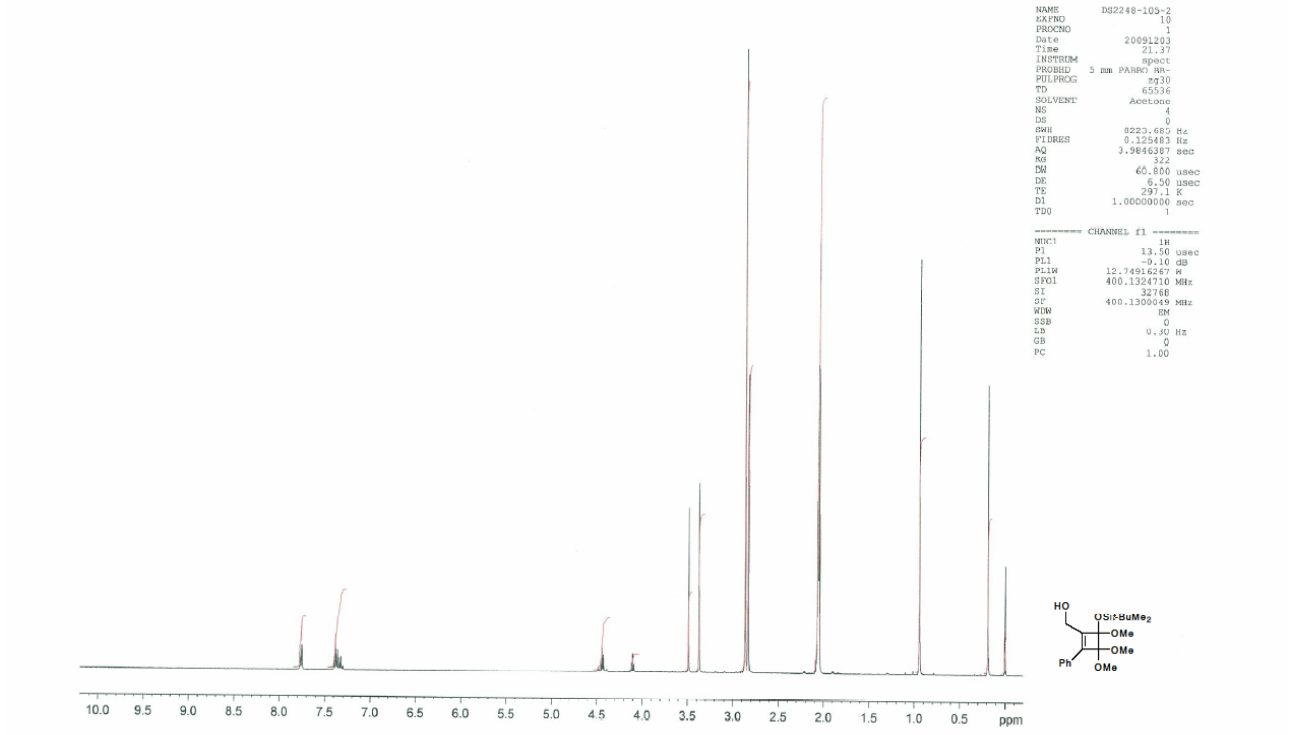

1H 785-1-13c
Date : Wed May 3 18:33:59 2006
FileName : e785-1-13c.nmdata
Comment : 1H 785-1-13c
SliceHistory :
EXMODE : dcm
POINT : 65536 points
SAMPO : 65536 points
FREQU : 26881.7 Hz
FILTR : 13450 Hz
DELAY : 14.9 usec
DEADT : 19.9 usec
INTVL : 37.2 usec
TIMES : 500 times
DUMMY : 1 times
PD : 1.5000 sec
ACQTM : 2437.9392 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.41 Hz
PW1 : 4.90 usec
OBNUC : 13C
OBFRQ : 99.45 MHz
OBSET : 104750.00 Hz
RGAIN : 34
IRNUC : 1H
IRFRQ : 395.75 MHz
IRSET : 134498.00 Hz
IRPPM : 46.0 usec
IRRNS : 0
SCANS : 500 times
SLVNT : ACETN
SPINNING : 11 Hz
TEMP : 24.4 C
OSi-BuMe2
PhO
OMe
OMe
PMBO
OMe
200
150
100
50
0
ppm

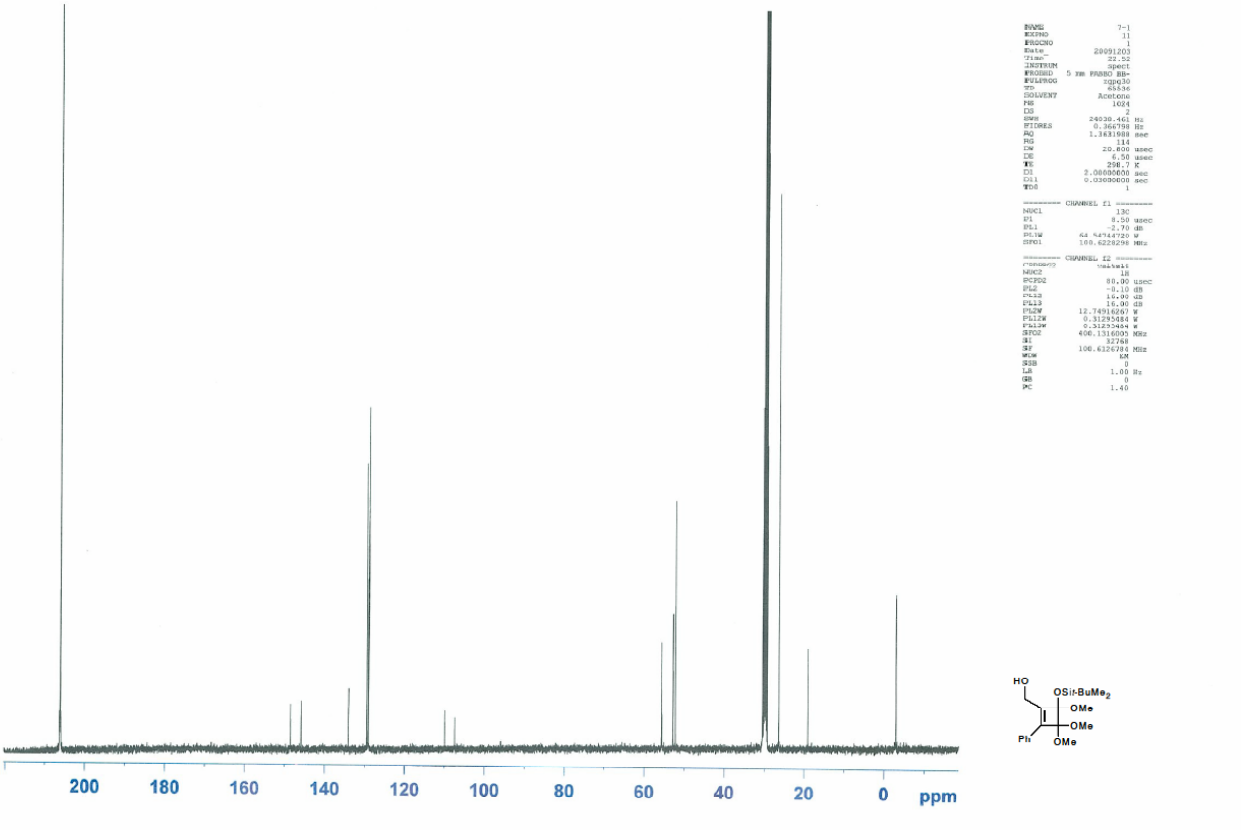

1H 061123-1
Date : Thu Nov 23 16:18:19 2006
FileName : LoadingFID.nmdat
Comment : 1H 061123-1
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7917.7 Hz
FILTR : 3950 Hz
DELAY : 50.6 usec
DEADT : 72.3 usec
INTVL : 126.3 usec
TIMES : 8 times
DUMMY : 1 times
PD : 2.9007 sec
ACQTM : 4138.5986 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.24 Hz
PW1 : 7.25 usec
OBNUC : 1H
OBFRQ : 395.75 MHz
OBSET : 134498.00 Hz
RGAIN : 23
SCANS : 8 times
SLVNT : ACETN
SPINNING : 10 Hz
TEMP : 24.5 C
10
8
6
4
2
0
ppm
PhO
OSi-tBuMe2
OMe
OMe
PMBO
OMe

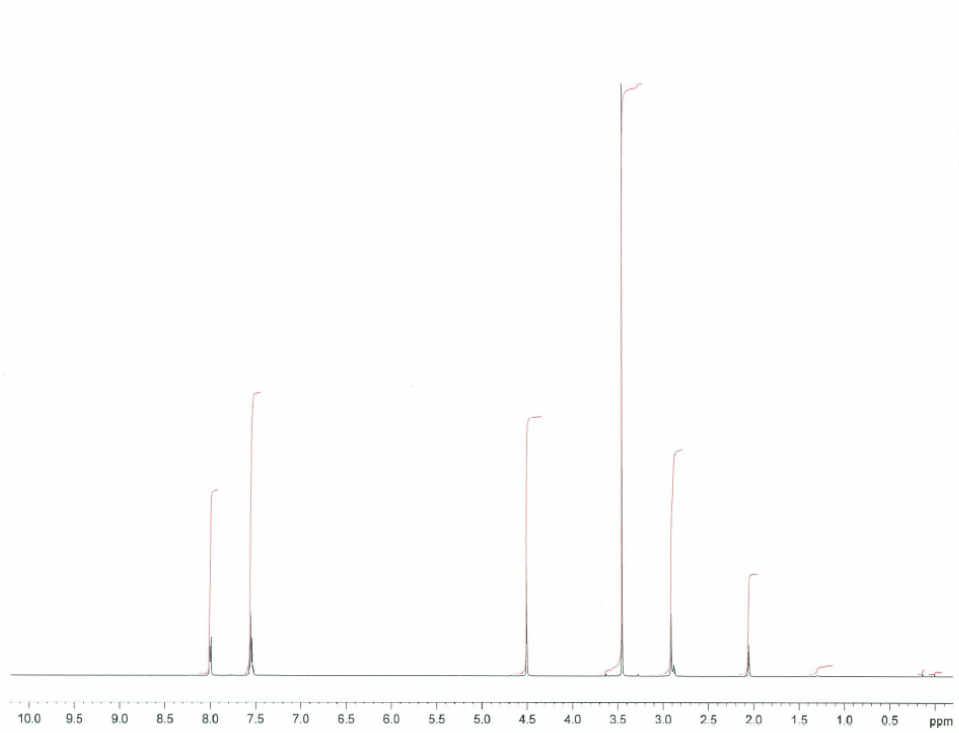

1H 061124-1-13c
Date : Fri Nov 24 13:15:44 2006
FileName : LoadingFID.nmdat
Comment : 1H 061124-1-13c
SliceHistory :
EXMODE : bcm
POINT : 65536 points
SAMPO : 65536 points
FREQU : 26881.7 Hz
FILTR : 13450 Hz
DELAY : 14.9 usec
DEADT : 19.9 usec
INTVL : 37.2 usec
TIMES : 1000 times
DUMMY : 1 times
PD : 1.5000 sec
ACQTM : 2437.9392 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.41 Hz
PW1 : 4.90 usec
OBNUC : 13C
OBFRQ : 99.45 MHz
OBSET : 104750.00 Hz
RGAIN : 35
IRNUC : 1H
IRFRQ : 395.75 MHz
IRSET : 134498.00 Hz
IRPPW : 50.0 usec
IRPNS : 0
SCANS : 1000 times
SLVNT : ACETN
SPINNING : 14 Hz
TEMP : 25.4 C
200
150
100
50
0
ppm
PhO
OSi-tBuMe2
OMe
OMe
PMBO
OMe

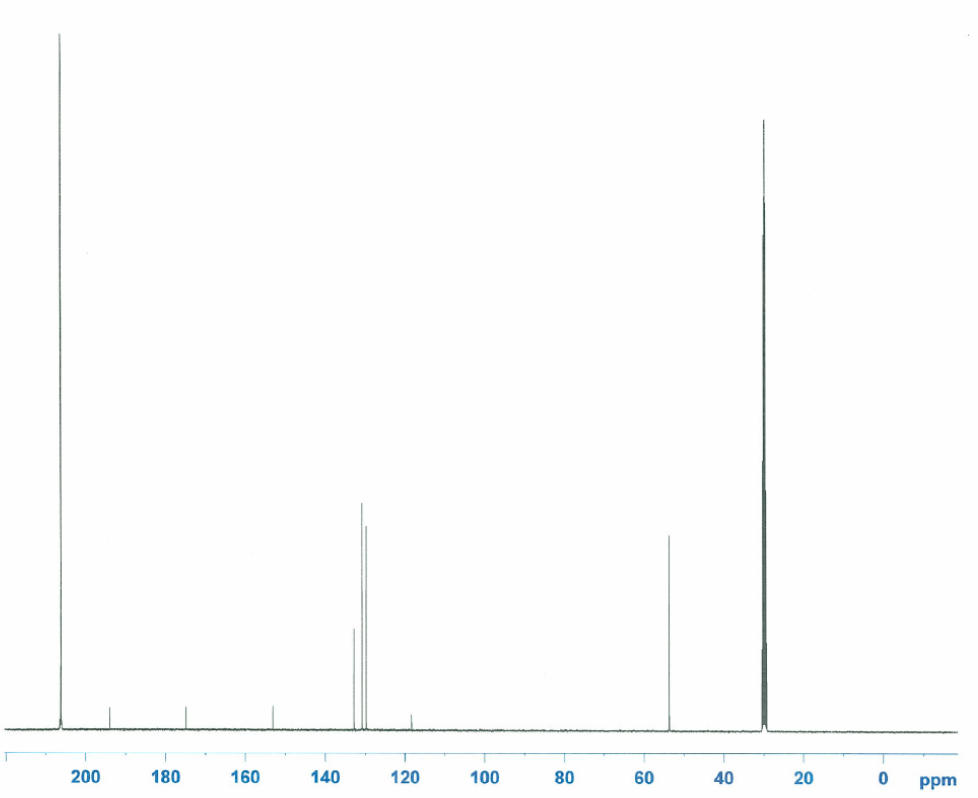

1H 795-2
Date : Thu May 11 16:55:41 2006
FileName : LoadingFID.nmdat
Comment : 1H 795-2
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7917.7 Hz
FILTR : 3950 Hz
DELAY : 50.6 usec
DEADT : 72.4 usec
INTVL : 126.3 usec
TIMES : 8 times
DUMMY : 1 times
PD : 2.8614 sec
ACQTM : 4138.5986 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.24 Hz
PW1 : 7.00 usec
OBNUC : 1H
OBFRQ : 395.75 MHz
OBSET : 134498.00 Hz
RGAIN : 23
SCANS : 8 times
SLVNT : ACETN
SPINNING : 13 Hz
TEMP : 24.2 C
PhO
O
O
OMe
Ph
OMe
10
8
6
4
2
0
ppm

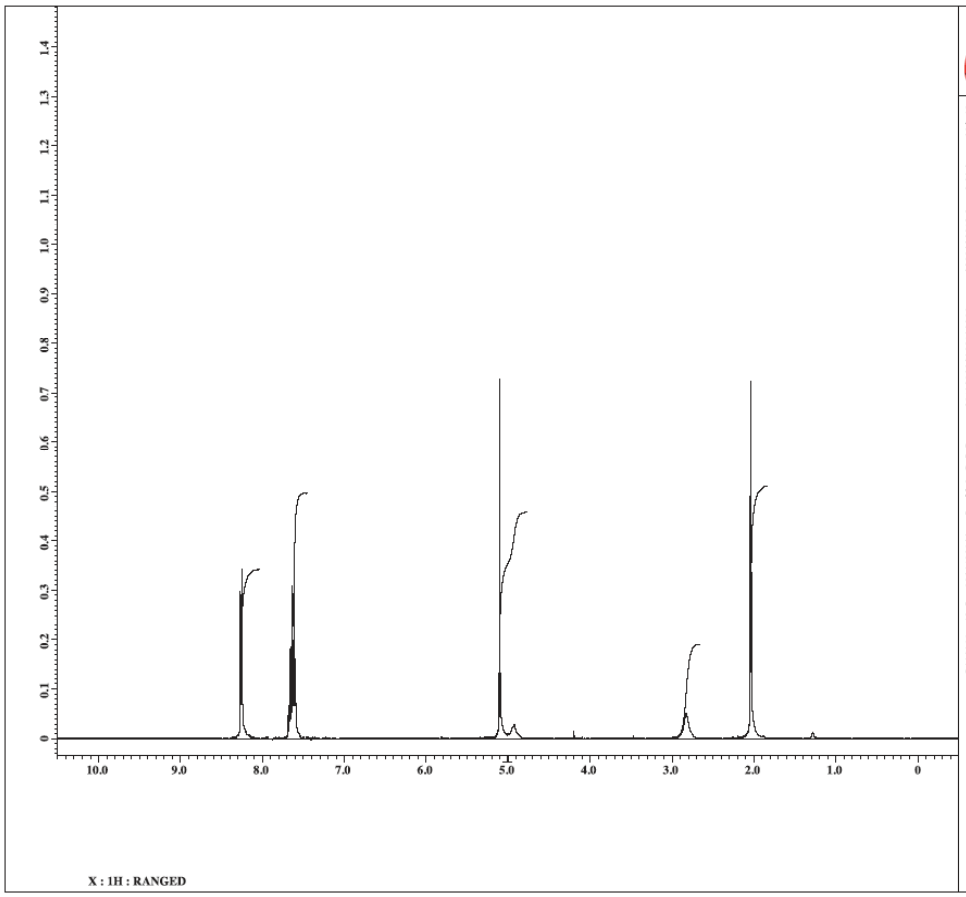

1H 795-2-13c
Date : Thu May 11 16:43:15 2006
FileName : LoadingFID.nmdat
Comment : 1H 795-2-13c
SliceHistory :
EXMODE : bcm
POINT : 65536 points
SAMPO : 65536 points
FREQU : 26881.7 Hz
FILTR : 13450 Hz
DELAY : 14.9 usec
DEADT : 19.9 usec
INTVL : 37.2 usec
TIMES : 1000 times
DUMMY : 1 times
PD : 1.5000 sec
ACQTM : 2437.9392 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.41 Hz
PW1 : 4.90 usec
OBNUC : 13C
OBFRQ : 99.45 MHz
OBSET : 104750.00 Hz
RGAIN : 35
IRNUC : 1H
IRFRQ : 395.75 MHz
IRSET : 134498.00 Hz
IRPPW : 46.0 usec
IRNS : 0
SCANS : 1000 times
SLVNT : ACETN
SPINNING : 13 Hz
TEMP : 25.0 C
PhO
O
O
OMe
Ph
OMe
200
150
100
50
0
ppm


NAME DS2248-105-2
EXPNO 10
PROCNO 1
Date 20091203
Time 21.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 4
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 322
DW 60.800 usec
DE 6.50 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.50 usec
PL1 -0.10 dB
PL1W 12.74916267 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300049 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
HO
OSi-t-BuMe2
OMe
OMe
Ph
OMe
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

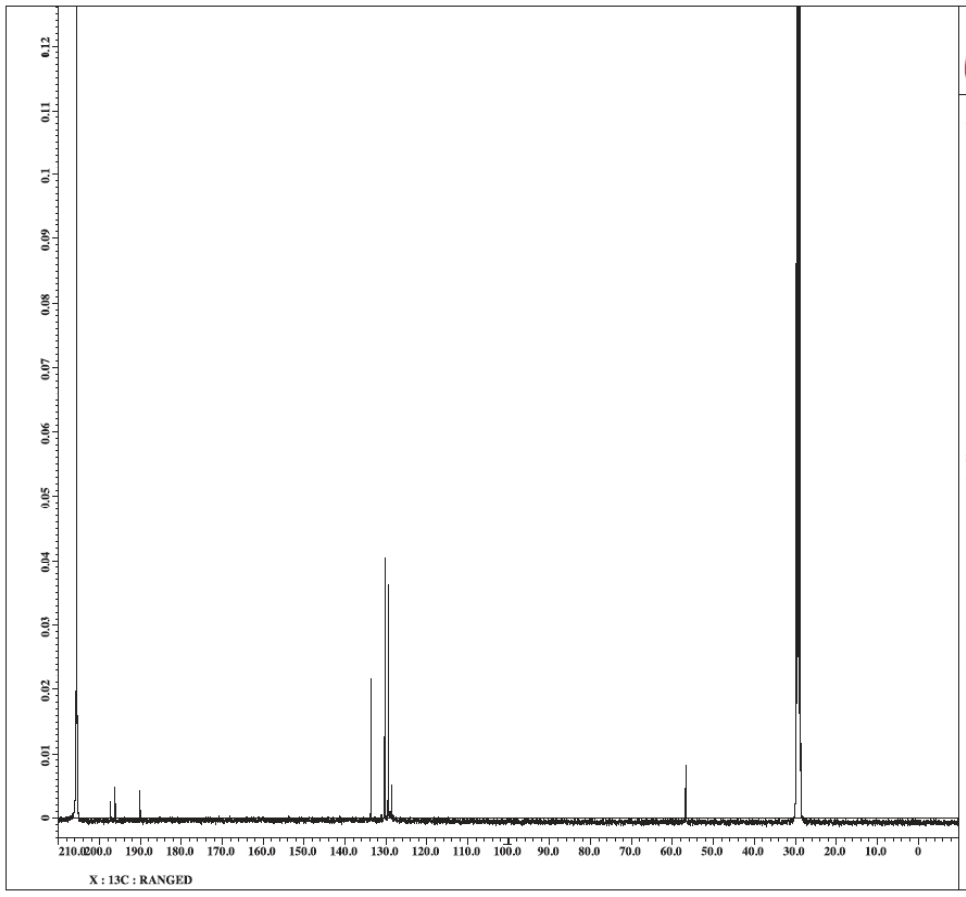

NAME 7-1
EXPNO 11
PROCNO 1
Date 20091203
Time 22.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 1024
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 114
DW 20.800 usec
DE 6.50 usec
TE 298.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 8.50 usec
PL1 -2.70 dB
PL1W 64.54744720 W
SFO1 100.6228298 MHz
CHANNEL f2
NUC2 1H
PCPD2 80.00 usec
PL2 -0.10 dB
PL12 16.90 dB
PL13 16.90 dB
PL2W 12.74916267 W
PL12W 0.31295484 W
PL13W 0.31295484 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6126796 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
HO
OSi-t-BuMe2
OMe
OMe
Ph
OMe
200 180 160 140 120 100 80 60 40 20 0 ppm


10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm


200 180 160 140 120 100 80 60 40 20 0 ppm




```
Filename            = 090-10-23hamura-iwata
Author              = delta
Experiment          = single_pulse.ex2
Sample_id           = hamu
Solvent             = ACETONE-D6
Creation_time       =  9-MAR-2010 17:14:27
Revision_time       =  9-MAR-2010 19:51:11
Current_time        =  9-MAR-2010 19:52:23

Comment             = single_pulse
Data_format         = 1D REAL
Dim_size            = 13107
Dim_title           = 1H
Dim_units           = [ppm]
Dimensions          = X
Site                = RCX400M
Spectrometer        = DELTA2_NMR

Field_strength      = 9.389766[T] (400[MHz]
X_acq_duration      = 2.18365952[s]
X_domain            = 1H
X_freq              = 399.78219838[MHz]
X_offset            = 5[ppm]
X_points            = 16384
X_prescans          = 1
X_resolution        = 0.45794685[Hz]
X_sweep             = 7.5030012[kHz]
Irr_domain          = 1H
Irr_freq            = 399.78219838[MHz]
Irr_offset          = 5[ppm]
Tri_domain          = 1H
Tri_freq            = 399.78219838[MHz]
Tri_offset          = 5[ppm]
Clipped             = FALSE
Mod_return          = 1
Scans               = 32
Total_scans         = 32

X_90_width          = 10.5[us]
X_acq_time          = 2.18365952[s]
X_angle             = 45[deg]
X_atn               = 1.4[dB]
X_pulse             = 5.25[us]
Irr_mode            = Off
Tri_mode            = Off
Dante_presat        = FALSE
Initial_wait        = 1[s]
Recvr_gain          = 38
Relaxation_delay    = 5[s]
Repetition_time     = 7.18365952[s]
Temp_get            = 23.2[dC]
```




```
Filename            = 090hamura-tC=c=9txKh
Author              = delta
Experiment          = single_pulse_dec
Sample_id           = hamura
Solvent             = ACETONE-D6
Creation_time       = 27-NOV-2009 07:36:46
Revision_time       = 23-JAN-2010 17:50:49
Current_time        = 23-JAN-2010 17:53:14

Comment             = single pulse decouple
Data_format         = 1D COMPLEX
Dim_size            = 26214
Dim_title           = 13C
Dim_units           = [ppm]
Dimensions          = X
Site                = RCX400M
Spectrometer        = DELTA2_NMR

Field_strength      = 9.389766[T] (400[MHz]
X_acq_duration      = 1.04333312[s]
X_domain            = 13C
X_freq              = 100.52530333[MHz]
X_offset            = 100[ppm]
X_points            = 32768
X_prescans          = 4
X_resolution        = 0.95846665[Hz]
X_sweep             = 31.40703518[kHz]
Irr_domain          = 1H
Irr_freq            = 399.78219838[MHz]
Irr_offset          = 5[ppm]
Clipped             = FALSE
Mod_return          = 1
Scans               = 11200
Total_scans         = 11200

X_90_width          = 9.2[us]
X_acq_time          = 1.04333312[s]
X_angle             = 30[deg]
X_atn               = 6.6[dB]
X_pulse             = 3.06666667[us]
Irr_atn_dec         = 22.2[dB]
Irr_atn_noe         = 22.2[dB]
Irr_noise           = WALTZ
Decoupling          = TRUE
Initial_wait        = 1[s]
Noe                 = TRUE
Noe_time            = 2[s]
Recvr_gain          = 50
Relaxation_delay    = 2[s]
Repetition_time     = 3.04333312[s]
Temp_get            = 25.5[dC]
```