

Solvent-free hydrosilylation of alkenes and alkynes using recyclable platinum on carbon nanotubes

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and Eric Doris

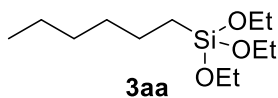
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General

Chemicals were purchased from Sigma-Aldrich and used as received. Electron microscopy observations were carried out on a Philips CM12 microscope operated at 100 kV. NMR spectra were recorded on a Bruker Avance spectrometer at 400 MHz (^1H), 100 MHz (^{13}C), and 61 MHz (^2H). Chemical shifts are given in ppm relative to the NMR solvent residual peak. ^{29}Si NMR experiments were performed at 298 K on a 500 MHz Avance II Bruker spectrometer equipped with an inverse broadband 5 mm probe head (^{29}Si NMR frequency: 99.362 MHz). Spectra were acquired with 64 scans and an interscan delay of 60 sec. Chemical shifts are calibrated considering the signal of triethylsilane at 0 ppm. XPS spectra were acquired on a VG ESCALAB 210 spectrometer. HRMS were recorded on a Waters Xevo G2-XS QToF spectrometer.

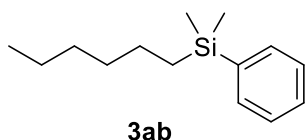
1. Spectroscopic data



$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.57 (t, 2H), 0.81 (t, 3H), 1.12–1.38 (m, 17H), 3.72–3.81 (m, 6H) ppm.¹

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 10.5, 14.3, 18.4, 22.7, 22.9, 31.6, 33.0, 58.4 ppm.

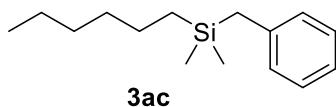
$^{29}\text{Si-NMR}$ (99.3 MHz, CDCl_3) δ 7.04 ppm.



$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.41 (s, 6H), 0.91 (t, 2H), 1.02 (t, 3H), 1.42–1.47 (m, 8H), 7.48 (m, 3H, ArH), 7.65 (m, 2H, ArH) ppm.²

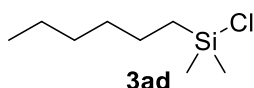
$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ -2.9, 14.3, 16.0, 22.8, 24.0, 31.7, 33.5, 127.8, 128.9, 133.7, 139.8 ppm.

$^{29}\text{Si-NMR}$ (99.3 MHz, CDCl_3) δ -3.23 ppm.



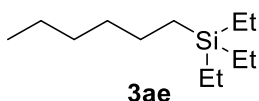
$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.00 (s, 6H), 0.54 (t, 2H), 0.93 (t, 3H), 1.32 (m, 8H), 2.12 (s, 2H), 7.11 (m, 3H, ArH), 7.24 (dd, 2H, ArH) ppm.³

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ -3.5, 14.3, 15.0, 22.7, 23.8, 25.7, 31.7, 33.4, 123.9, 128.1, 128.2, 140.6 ppm.



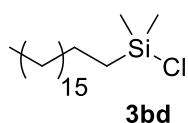
$^1\text{H-NMR}$ (400 MHz, CDCl_3): δ 0.40 (s, 6H), 0.80 (t, 2H), 0.91 (t, 3H), 1.24–1.45 (m, 8H) ppm.⁴

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3 , ppm) δ 1.8, 14.3, 19.2, 22.7, 23.1, 31.6, 32.8 ppm.



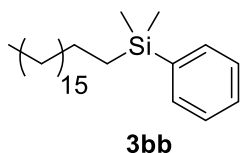
$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.47–0.53 (m, 8H), 0.87–0.98 (m, 12H), 1.23–1.34 (m, 8H) ppm.⁵

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 3.5, 7.6, 11.5, 14.3, 22.8, 24.0, 32.0, 33.8 ppm.



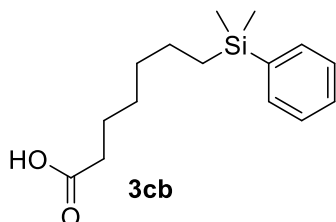
$^1\text{H-NMR}$ (400 MHz, CDCl_3 , ppm) δ 0.41 (s, 6H), 0.88, (t, 2H), 0.92 (t, 3H), 1.23–1.46 (m, 32H) ppm.

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3 , ppm) δ 1.8, 14.3, 19.2, 22.9, 23.2, 29.5, 29.6, 29.8, 29.9, 30.0, 32.2, 33.2 ppm.



$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.37 (s, 6H), 0.87, (t, 2H), 1.0 (t, 3H), 1.39–1.55 (m, 32H), 7.44 (m, 3H, ArH), 7.61 (m, 2H, ArH) ppm.⁶

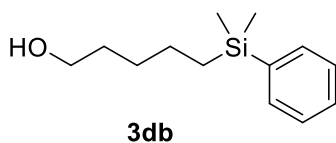
$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ -2.9, 14.3, 15.9, 22.9, 24.0, 27.1, 29.5, 29.6, 28.8, 29.9, 32.1, 33.8, 128.0, 128.8, 133.6, 139.8 ppm.



$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.27 (s, 6H), 0.76, (t, 2H), 1.28–1.38 (m, 6H), 1.61 (m, 2H), 2.34 (t, 2H), 7.36 (m, 3H, ArH), 7.52 (m, 2H, ArH) ppm.

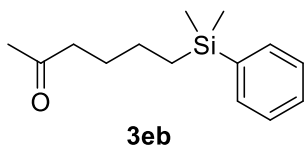
$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ -3.0, 15.7, 23.7, 24.6, 28.7, 33.2, 34.1, 127.7, 128.8, 133.6, 139.6, 180.4 ppm.

HRESMS m/z 263.1465 (calcd for $\text{C}_{15}\text{H}_{23}\text{O}_2\text{Si}$, 263.1467 [$\text{M} - \text{H}$]⁻).



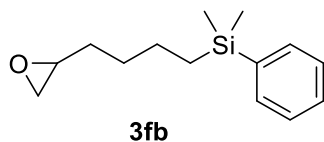
$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.26 (s, 6H), 0.76 (t, 2H), 1.24–1.37 (m, 4H), 1.51–1.62 (m, 2H), 3.60 (t, 2H), 7.35 (m, 3H, ArH), 7.50 (m, 2H, ArH) ppm.⁷

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ -3.3, 15.7, 23.7, 29.6, 32.5, 63.0, 127.7, 128.8, 133.6, 139.6 ppm.



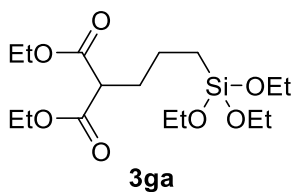
$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.15 (s, 6H), 0.64, (t, 2H), 1.21 (m, 2H), 1.47 (m, 2H), 1.95 (s, 3H), 2.25 (t, 3H), 7.22 (m, 3H, ArH), 7.38 (m, 2H, ArH) ppm.⁸

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ -3.1, 15.5, 23.5, 27.5, 29.7, 43.3, 127.7, 128.7, 133.4, 139.2 ppm.



$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.36 (s, 6H), 0.86, (t, 2H), 1.43–1.57 (m, 6H), 2.47 (q, 1H), 2.76 (t, 1H), 2.92 (q, 1H), 7.42 (m, 3H, ArH), 7.52 (dd, 2H, ArH) ppm.⁹

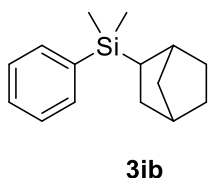
$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ -3.1, 15.63, 23.7, 29.7, 32.0, 46.9, 52.1, 127.7, 128.7, 133.4, 139.3 ppm.



$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.66 (t, 2H), 1.21–1.29 (m, 15H), 1.47 (m, 2H), 1.94 (q, 2H), 3.36 (t, 1H), 3.83 (m, 6H), 4.19 (q, 4H) ppm.¹⁰

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 10.1, 14.1, 18.3, 20.8, 32.0, 51.8, 58.4, 61.3, 169.5 ppm.

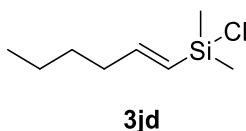
HRESMS m/z 387.1789 (calcd for $\text{C}_{16}\text{H}_{32}\text{NaO}_7\text{Si}$, 387.1815 $[\text{M} + \text{Na}]^+$).



$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.26 (s, 3H), 0.28 (s, 3H), 0.85 (m, 1H), 1.09–1.12 (m, 2H), 1.21–1.29 (m, 2H), 1.40–1.59 (m, 4H), 2.25 (m, 2H), 7.36–7.38 (m, 3H, ArH), 7.53–7.56 (m, 2H, ArH) ppm.¹¹

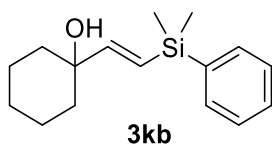
$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ -4.1, -4.0, 28.6, 28.9, 32.8, 34.4, 37.0, 37.8, 38.0, 127.7, 128.7, 134.0, 139.4 ppm.

$^{29}\text{Si-NMR}$ (99.3 MHz, CDCl_3) δ -3.38 ppm.



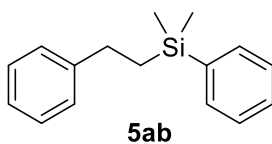
$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.36 (s, 6H), 0.80 (t, 3H), 1.21–1.30 (m, 4H), 2.05 (m, 2H), 5.61 (d, 1H), 6.16 (m, 1H) ppm.¹²

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 2.1, 14.1, 22.4, 33.6, 36.2, 126.1, 151.3 ppm.



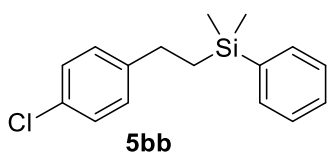
$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.34 (s, 6H), 0.74 (t, 4H), 0.84–1.23–1.35 (m, 2H), 1.49–1.71 (m, 10H), 6.03 (d, 1H), 6.24 (d, 1H), 7.33–7.36 (m, 3H, ArH), 7.50–7.52 (m, 2H, ArH) ppm.¹³

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ -2.3, 22.1, 25.7, 37.5, 72.8, 123.3, 127.9, 129.1, 134.0, 139.0, 155.4 ppm.



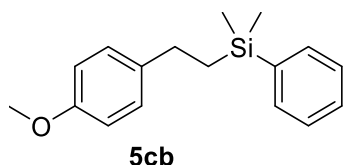
$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.50 (s, 6H), 1.34 (m, 2H), 2.85 (m, 2H), 7.38 (m, 3H, ArH), 7.47 (m, 2H, ArH), 7.56 (m, 3H, ArH), 7.75 (m, 2H, ArH) ppm.¹⁴

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ -2.9, 17.8, 30.1, 125.7, 127.9, 127.9, 128.4, 129.1, 133.7, 139.1, 145.1 ppm.



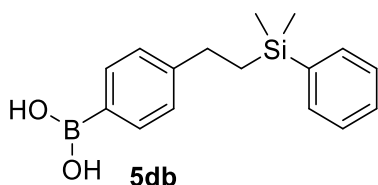
$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.50 (s, 6H), 1.28 (m, 2H), 2.79 (m, 2H), 7.24 (d, 2H, ArH), 7.41 (d, 2H, ArH), 7.56 (m, 3H, ArH), 7.74 (m, 2H, ArH) ppm.¹⁵

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ -3.0, 17.8, 29.5, 128.0, 128.4, 129.1, 129.4, 131.3, 133.7, 138.8, 143.4 ppm.



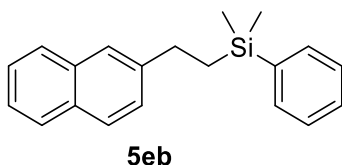
$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.35 (s, 6H), 1.16 (m, 2H), 2.65 (m, 2H), 3.83 (s, 3H), 6.87 (d, 2H, ArH), 7.14 (m, 2H, ArH), 7.41 (m, 3H, ArH), 7.58 (m, 2H, ArH) ppm.¹⁶

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ -3.0, 18.0, 29.1, 55.3, 113.7, 127.9, 128.7, 129.0, 133.7, 137.1, 139.2, 157.6 ppm.



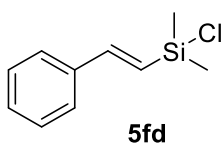
$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.45 (s, 6H), 1.29 (m, 2H), 2.82 (m, 2H), 7.39 (d, 2H, ArH), 7.51 (m, 2H, ArH), 7.68 (m, 2H, ArH), 8.24 (m, 2H, ArH) ppm.

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ -2.9, 17.6, 30.5, 127.6, 128.0, 129.1, 133.7, 135.9, 139.0, 150.0 ppm.



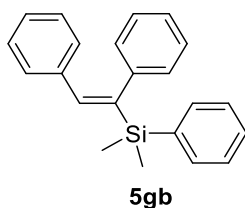
$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.41 (s, 6H), 1.30 (m, 2H), 2.88 (m, 2H), 7.39 (d, 1H, ArH), 7.44–7.53 (m, 5H, ArH), 7.63–7.67 (m, 3H, ArH), 7.81–7.87 (m, 3H, ArH) ppm.¹⁷

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ -2.9, 17.7, 30.2, 125.1, 125.5, 125.9, 127.1, 127.5, 127.7, 127.9, 129.0, 132.0, 133.7, 133.8, 139.1, 142.6 ppm.



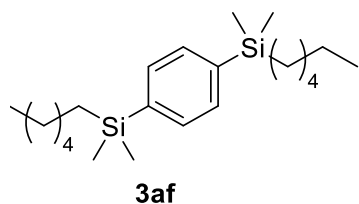
$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.66 (s, 6H), 6.51 (d, 1H), 7.13 (d, 1H), 7.42 (m, 3H, ArH), 7.53 (dd, 2H, ArH) ppm.¹⁸

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 2.2, 124.7, 127.0, 128.8, 129.0, 137.3, 146.7 ppm.



$^1\text{H-NMR}$ (400 MHz, CDCl_3 , ppm): δ 0.39 (s, 6H), 6.82 (s, 1H), 6.88-6.96 (m, 4H, ArH), 7.05-7.10 (m, 3H, ArH), 7.14-7.25 (m, 3H, ArH), 7.33-7.38 (m, 3H, ArH), 7.52-7.58 (m, 2H, ArH).¹⁹

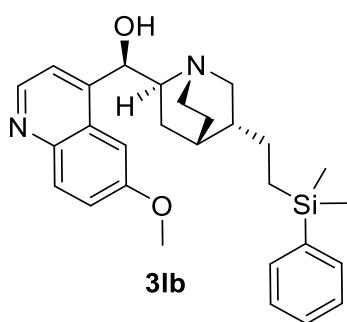
$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ -3.0, 125.7, 127.2, 127.7, 127.8, 127.9, 128.6, 129.1, 129.6, 134.3, 137.2, 137.7, 139.2, 142.3, 145.0 ppm.



$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ -0.24 (s, 12H), 0.74 (t, 4H), 0.84–0.88 (m, 6H), 1.24–1.32 (m, 16H), 7.49–7.51 (s, 4H) ppm.²⁰

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ -3.0, 14.1, 15.6, 22.6, 23.8, 31.5, 33.3, 132.8, 140.2 ppm.

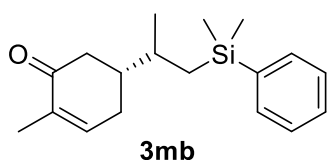
$^{29}\text{Si-NMR}$ (99.3 MHz, CDCl_3) δ -3.40 ppm.



$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.20 (s, 6H), 0.64 (t, 2H), 1.21–1.27 (m, 2H), 1.40–1.45 (m, 2H), 1.64–1.80 (m, 3H), 2.35 (d, 1H), 2.63 (d, 2H), 3.02–3.13 (m, 2H), 3.32–3.35 (m, 1H), 3.90 (s, 3H), 5.50 (d, 2H), 7.25–7.37 (m, 5H, ArH), 7.42 (d, 1H, ArH), 7.44 (d, 2H, ArH), 7.50 (d, 2H, ArH), 8.03 (d, 2H, ArH), 8.73 (d, 2H, ArH) ppm.

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ -3.2, 13.5, 21.8, 25.3, 28.3, 28.7, 38.8, 43.3, 55.7, 58.7, 59.8, 72.3, 101.3, 118.4, 121.6, 126.7, 127.7, 128.9, 131.8, 133.4, 139.2, 144.4, 147.2, 147.7, 157.8 ppm.

HRESMS m/z 461.2630 (calcd for $\text{C}_{28}\text{H}_{37}\text{N}_2\text{O}_2\text{Si}$, 461.2624 for $[\text{M} + \text{H}]^+$).

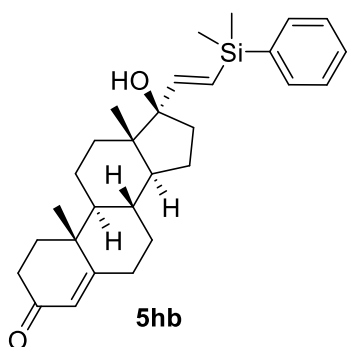


$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.30 (s, 6H), 0.61 (m, 1H), 0.86 (t, 3H), 0.93 (m, 1H), 1.62 (m, 1H), 1.75 (s, 3H), 1.79–2.47 (m, 5H), 6.69 (m, 1H), 7.34–7.36 (m, 3H, ArH), 7.48–7.51 (m, 2H, ArH) ppm.

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ -2.2, 15.8, 19.2, 20.5, 29.0, 29.7, 33.3, 33.4, 41.2, 42.0, 43.3, 43.3, 127.9, 129.0, 133.5, 135.2, 139.5, 145.4, 200.8 ppm.

$^{29}\text{Si-NMR}$ (99.3 MHz, CDCl_3) δ -3.50 ppm.

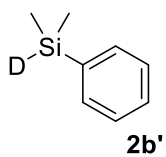
HRESMS m/z 309.1658 (calcd for $\text{C}_{18}\text{H}_{26}\text{NaOSi}$, 309.1651 $[\text{M} + \text{Na}]^+$).



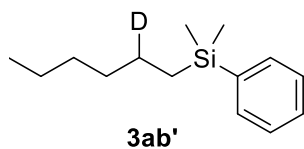
$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 0.36 (s, 6H), 0.82 (m, 1H), 0.96 (s, 3H), 1.01–1.17 (m, 2H), 0.96 (s, 3H), 1.19 (s, 3H), 1.24 (m, 2H), 1.35–1.56 (m, 4H), 1.59 (m, 2H), 1.65–1.69 (m, 3H), 1.81–1.88 (m, 2H), 1.95–2.0 (m, 2H), 2.05 (s, 1H), 2.25–2.44 (m, 3H), 5.73 (s, 1H), 5.86 (d, 1H), 6.24 (d, 1H), 7.31–7.37 (m, 3H, ArH), 7.49–7.52 (m, 2H, ArH) ppm.

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ -2.3, 14.4, 17.5, 20.8, 23.9, 31.8, 32.3, 34.1, 35.9, 36.2, 36.4, 38.8, 46.7, 49.8, 53.9, 85.0, 124.0, 124.6, 127.9, 129.2, 133.9, 138.9, 152.3, 171.3, 199.7 ppm.

HRESMS m/z 447.2714 (calcd for $\text{C}_{29}\text{H}_{39}\text{O}_2\text{Si}$, 447.2719 $[\text{M} - \text{H}]^-$).



¹H-NMR (400 MHz, CDCl₃) δ 0.38 (s, 6H), 7.37–7.44 (m, 3H, ArH), 7.59–7.61 (m, 2H, ArH) ppm.²¹



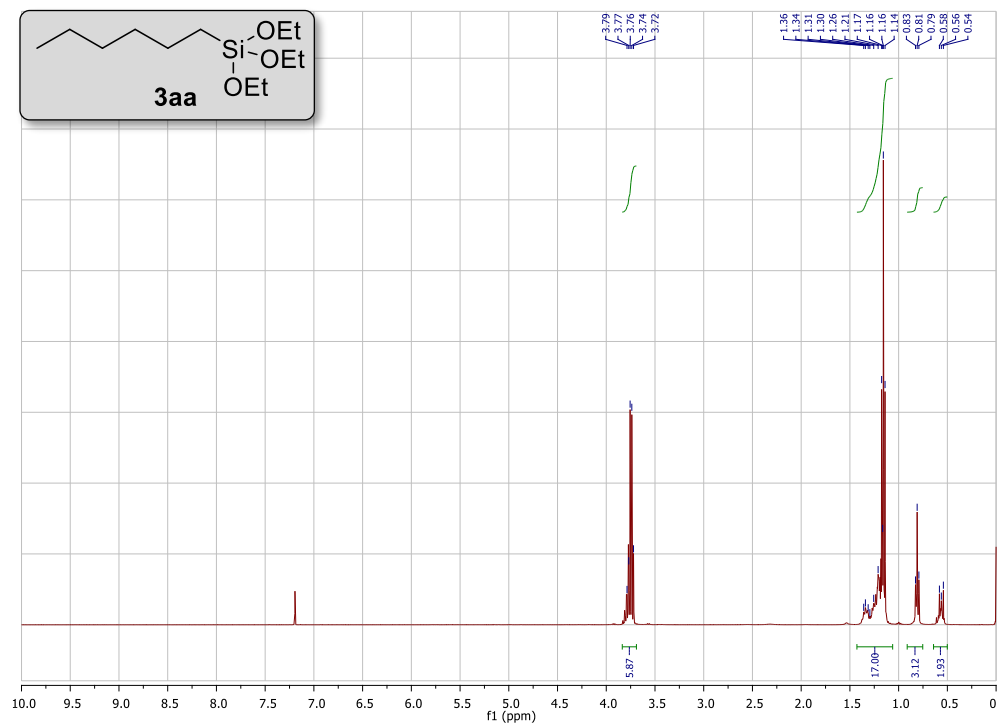
¹H-NMR (400 MHz, CDCl₃) δ 0.27 (s, 6H), 0.76 (d, 2H), 0.90 (m, 3H), 1.24–1.31 (m, 7H), 7.35–7.38 (m, 3H, ArH), 7.52 (m, 2H, ArH) ppm.

¹³C NMR (100 MHz, CDCl₃) δ -3.0, 14.1, 15.6, 22.6, 23.3 (t), 31.5, 33.2, 127.7, 128.7, 133.6, 139.8 ppm.

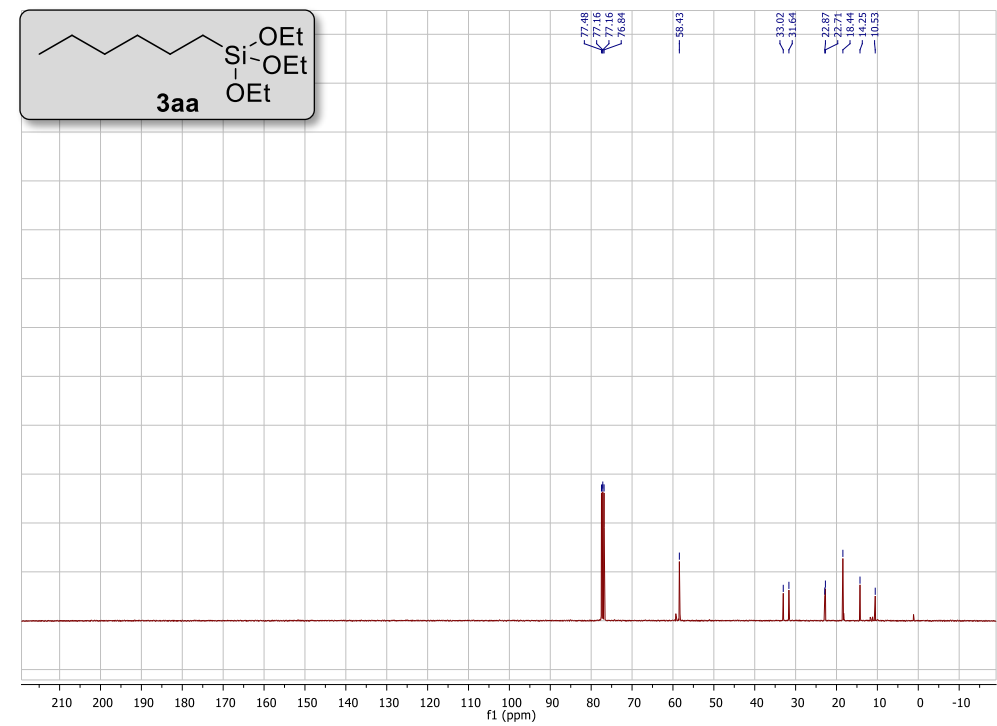
²H-NMR (61 MHz, CHCl₃) δ 1.27 ppm.

2. Copies of NMR spectra

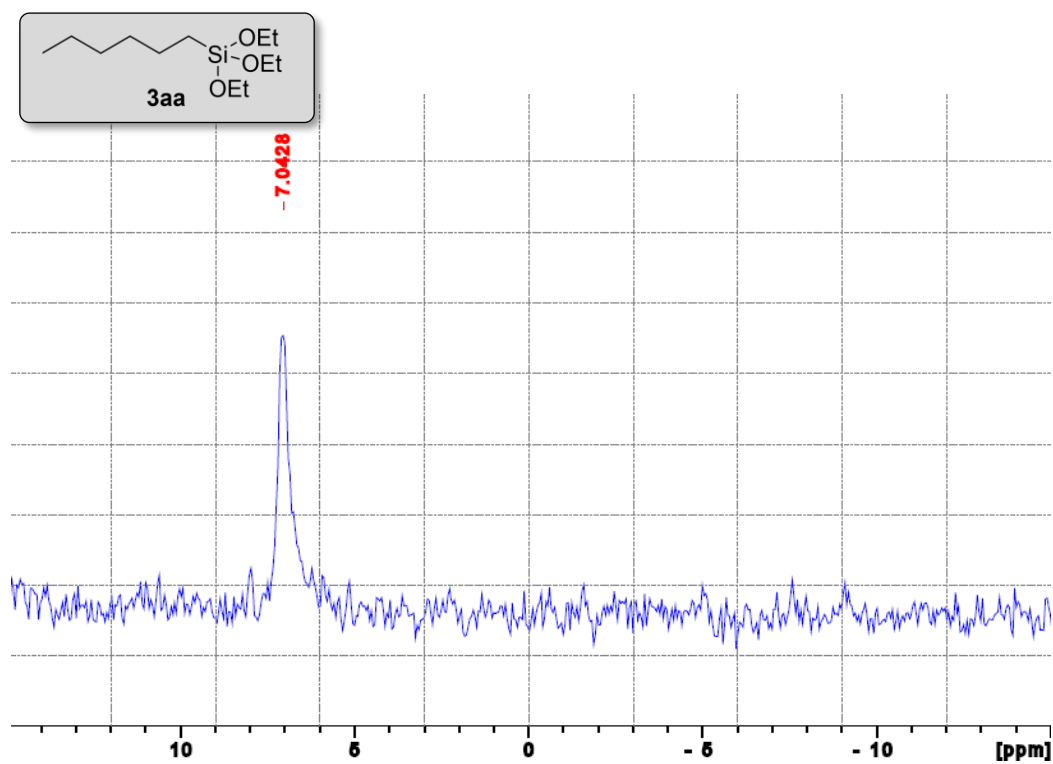
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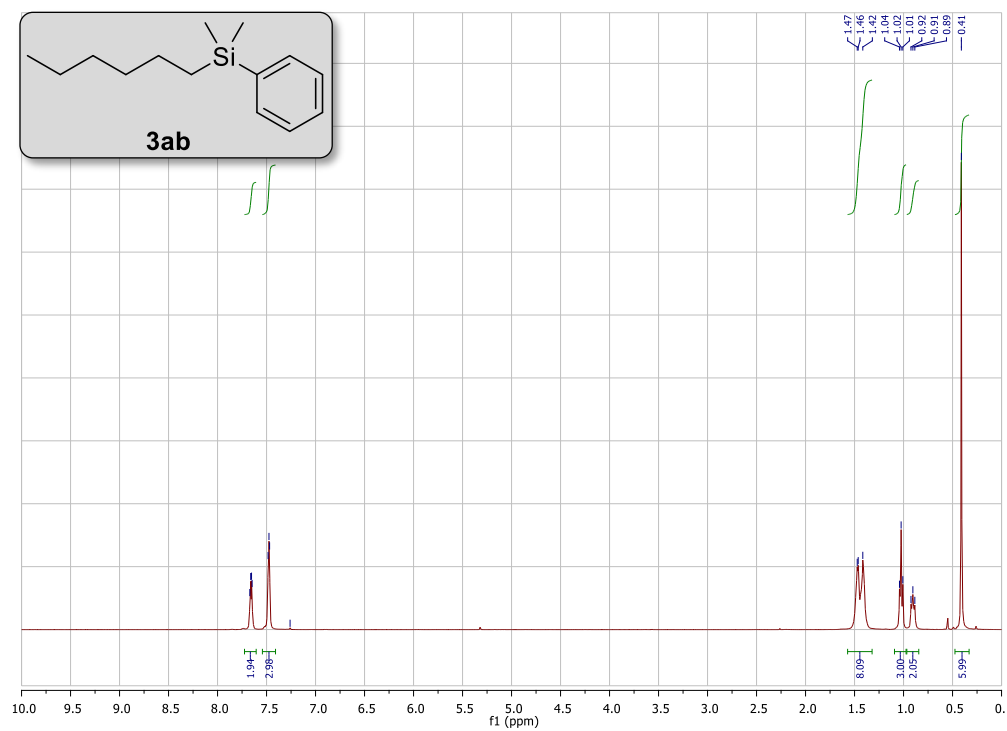
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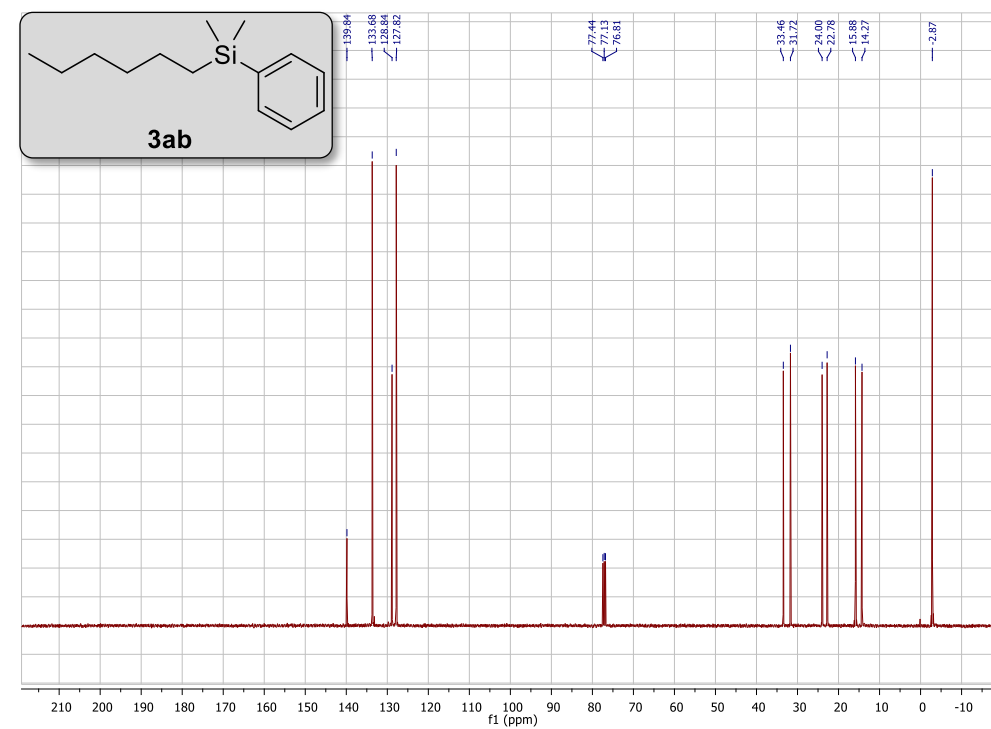
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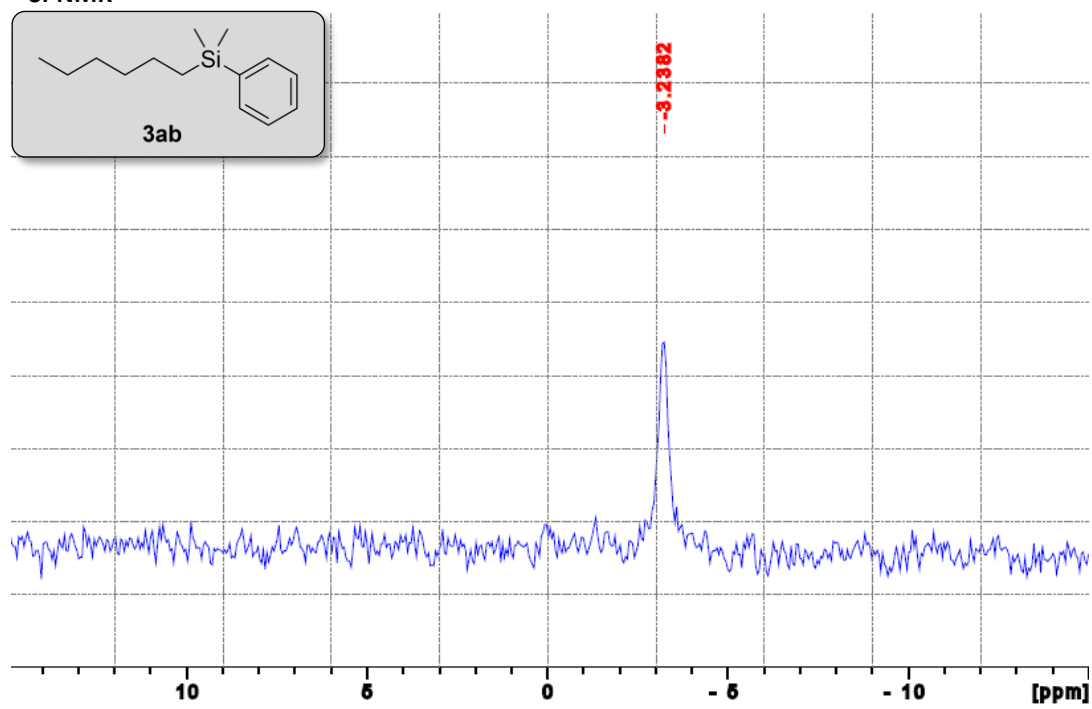
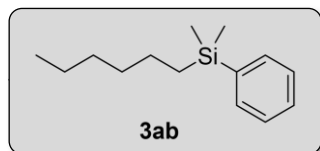
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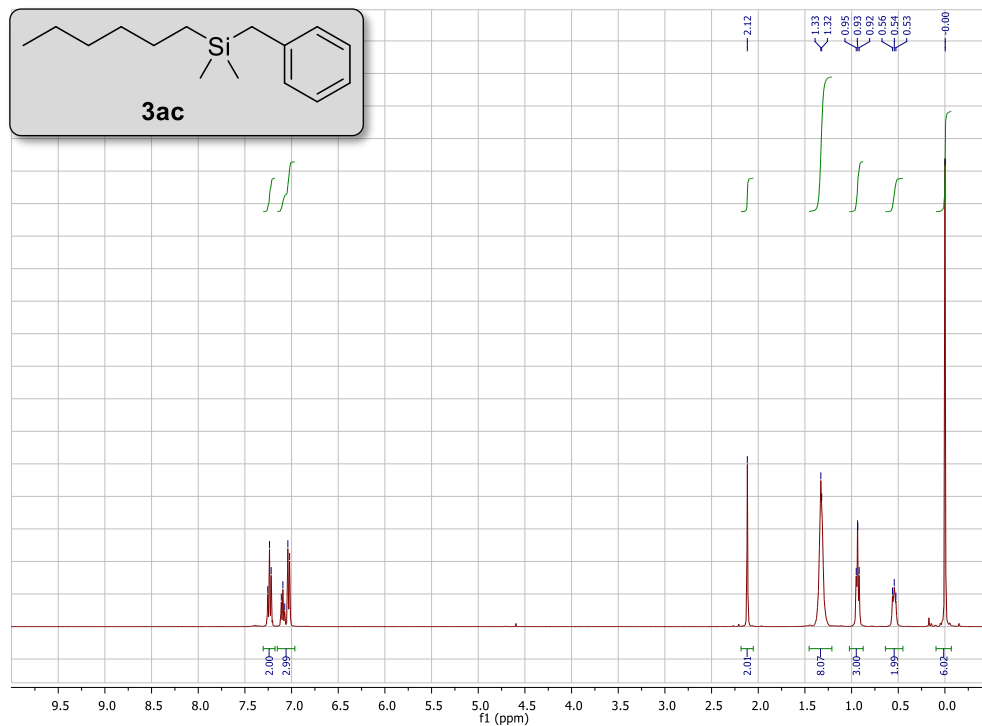
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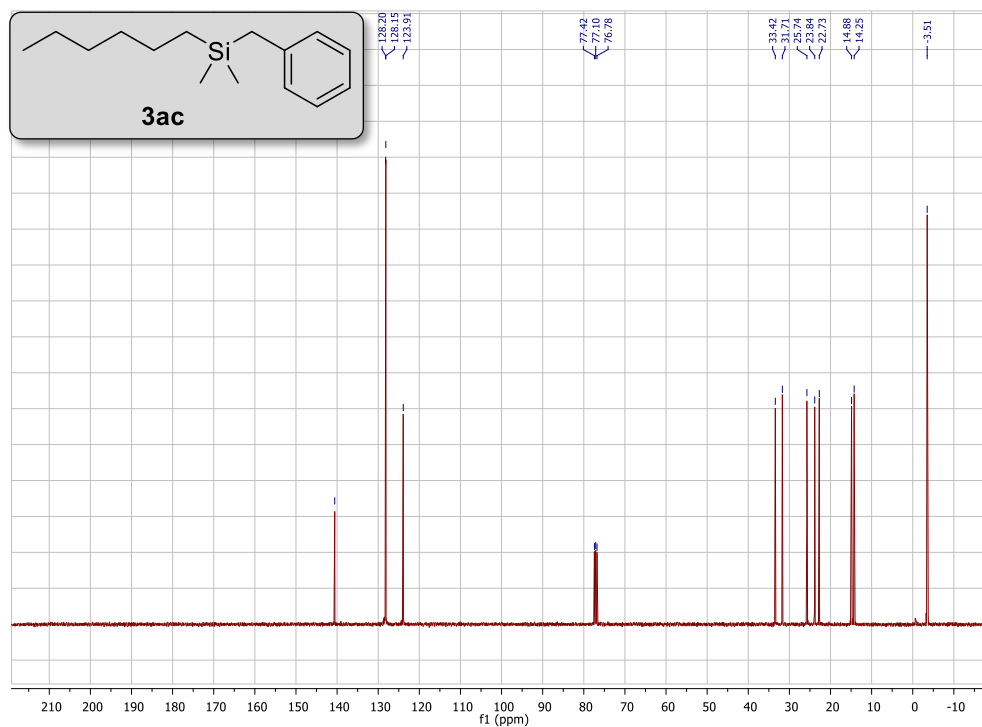
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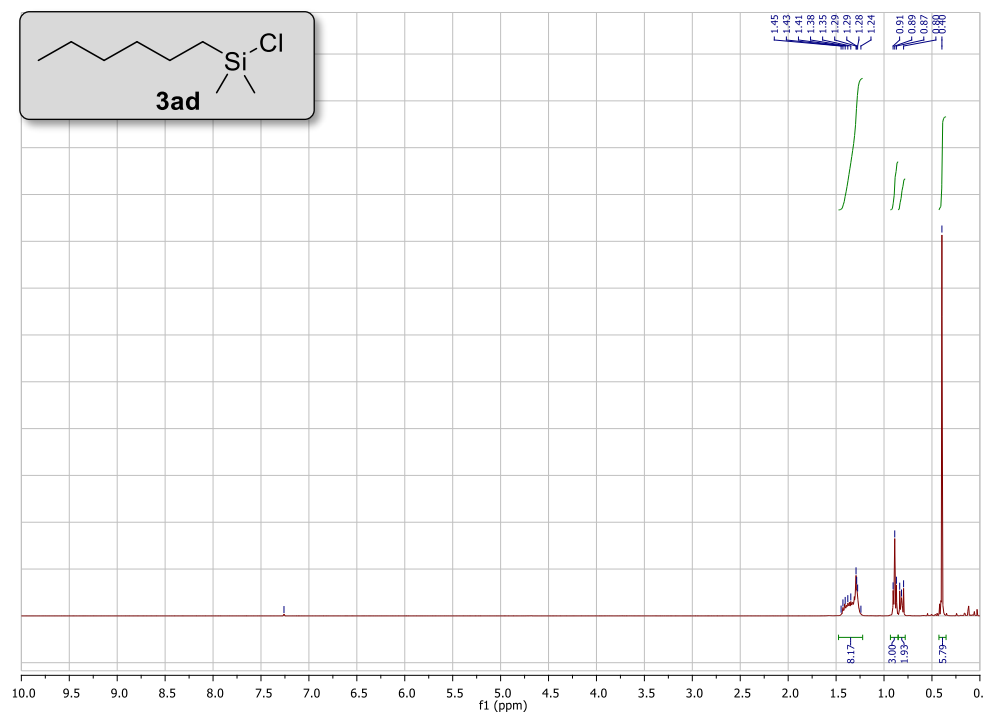
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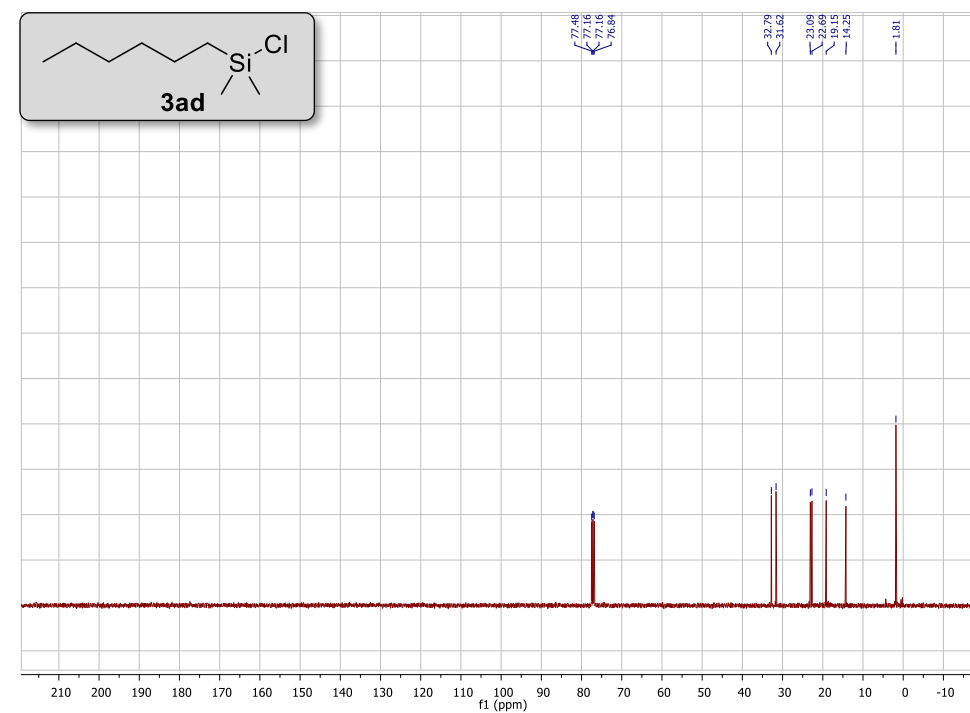
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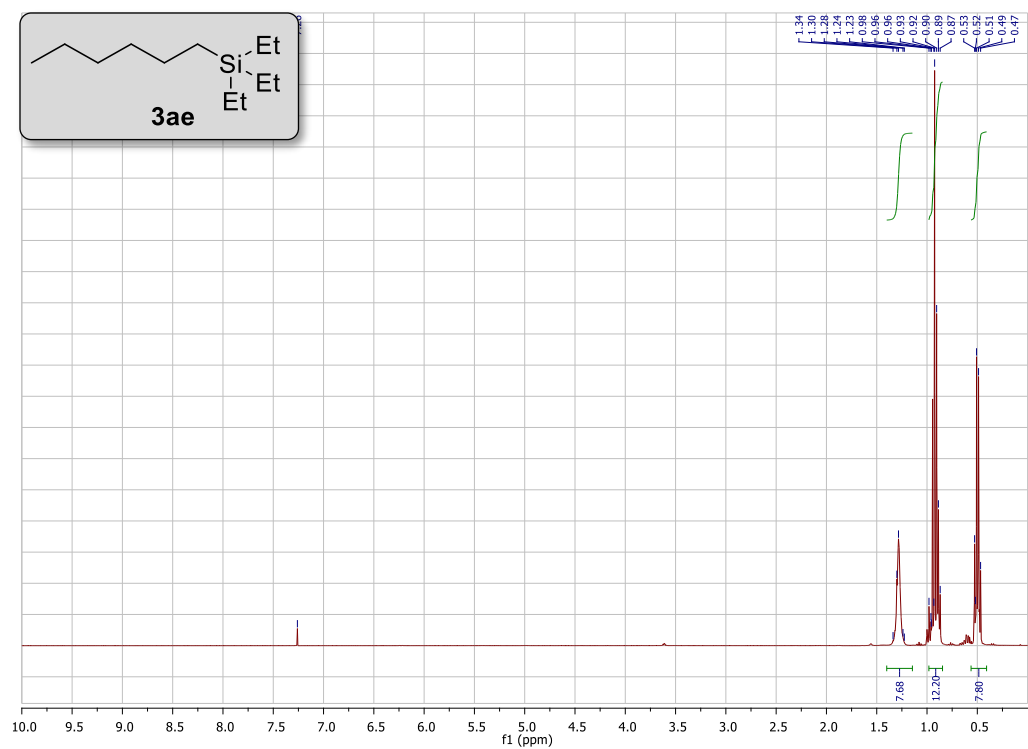
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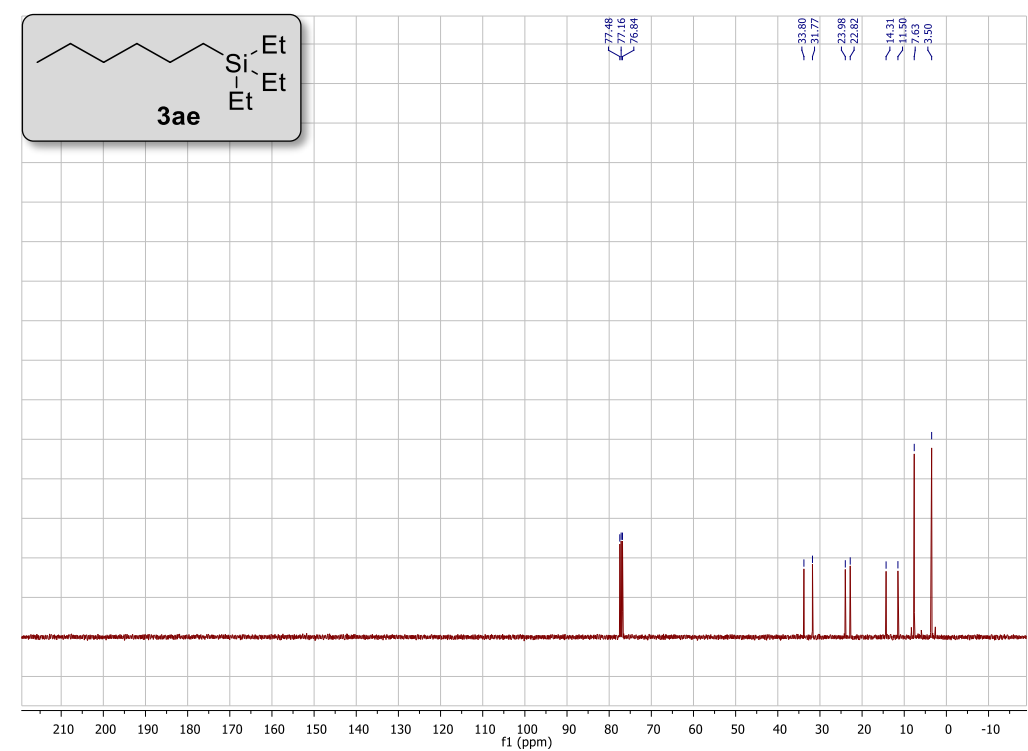
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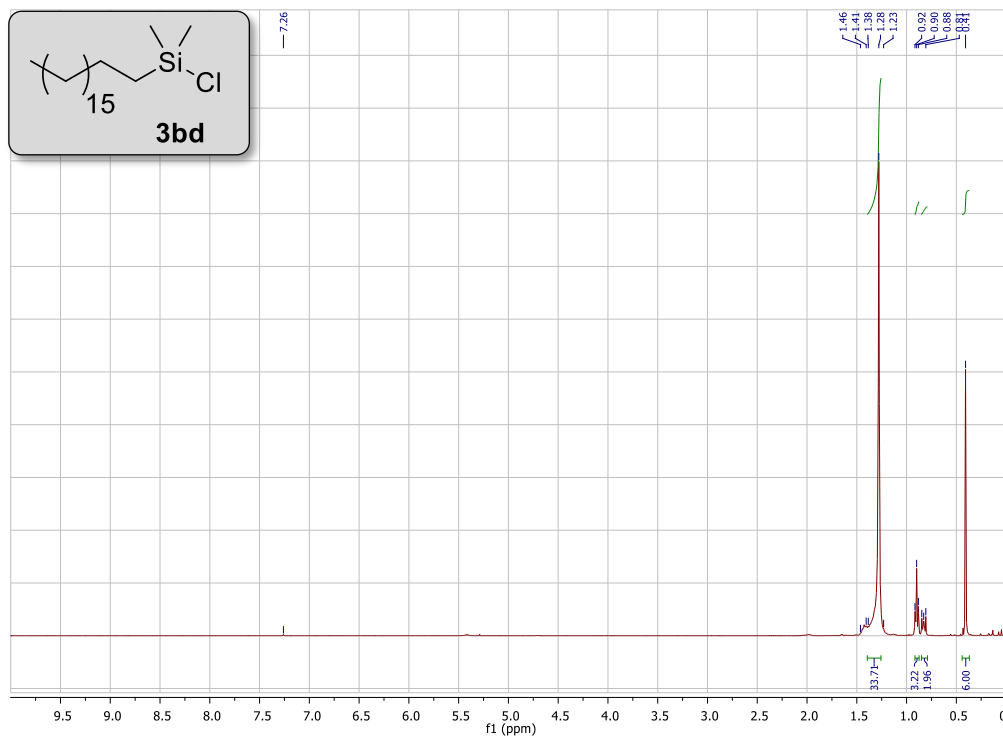
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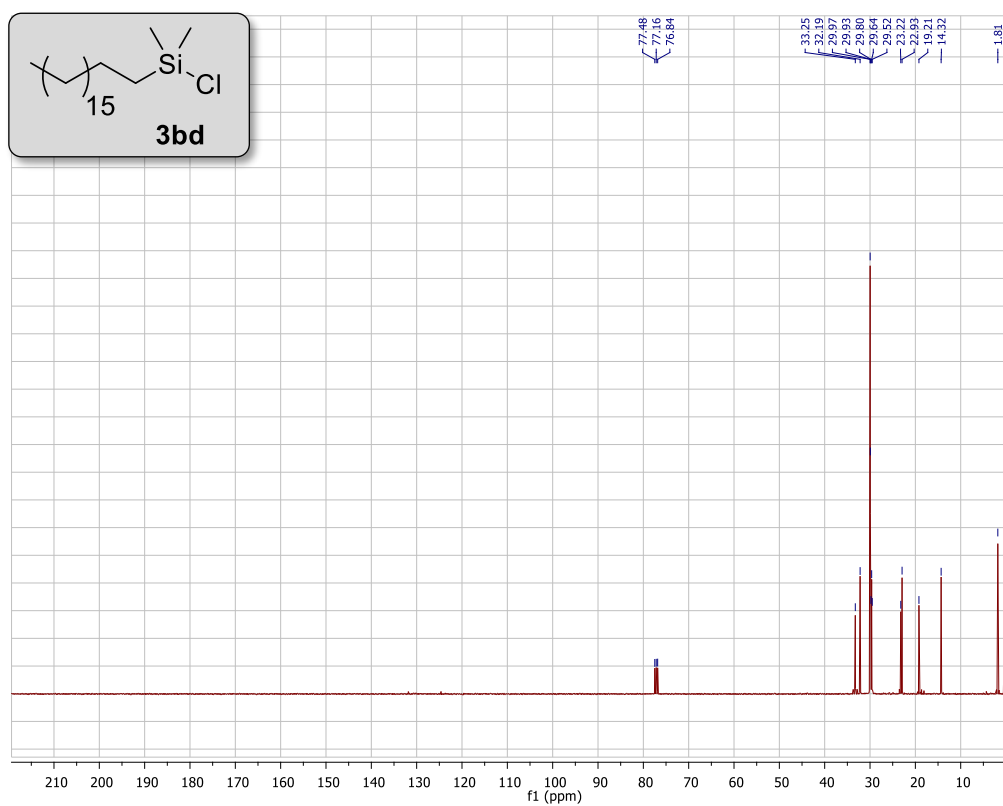
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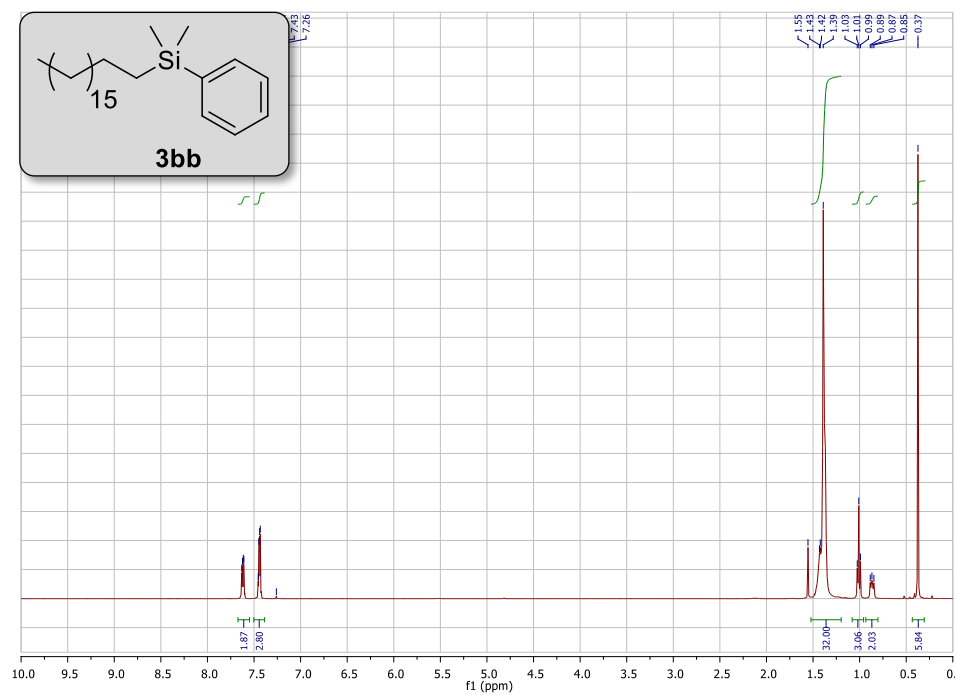
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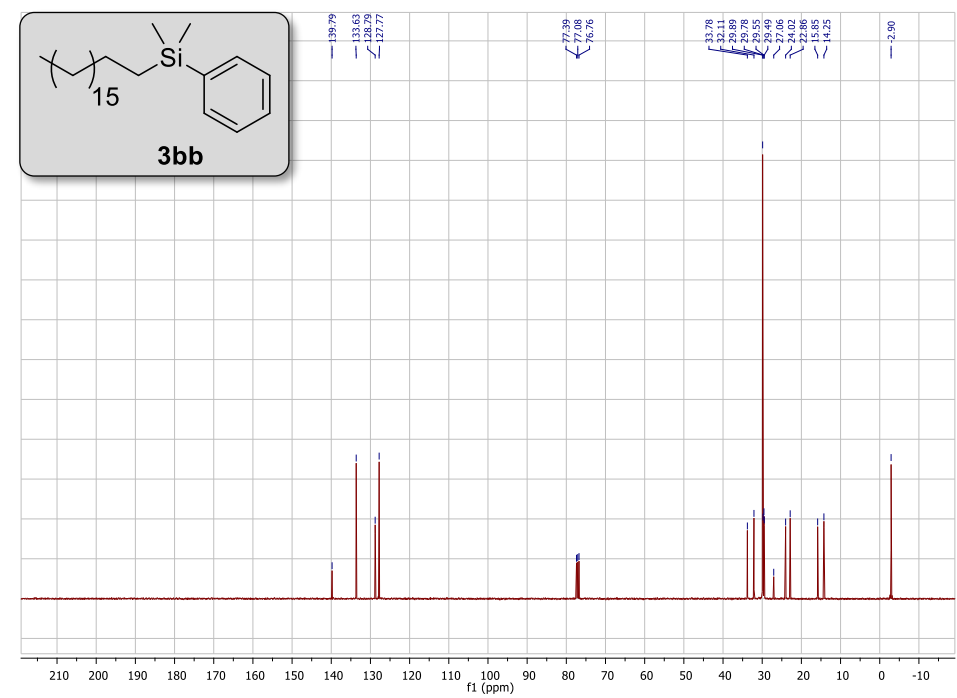
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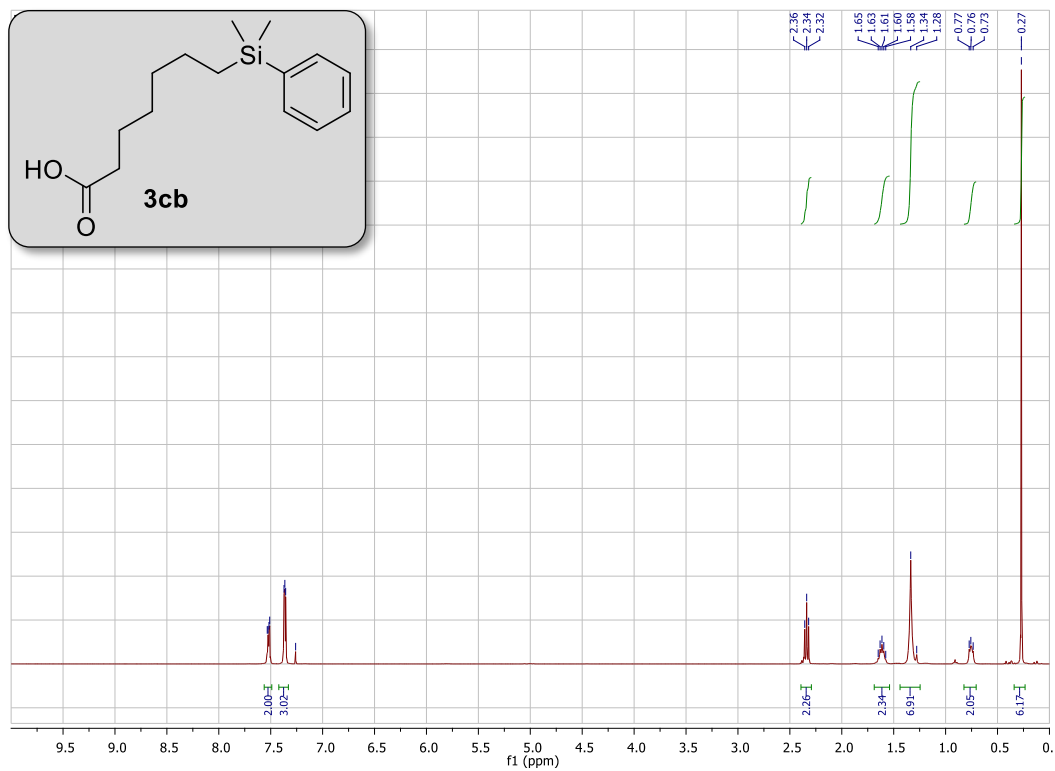
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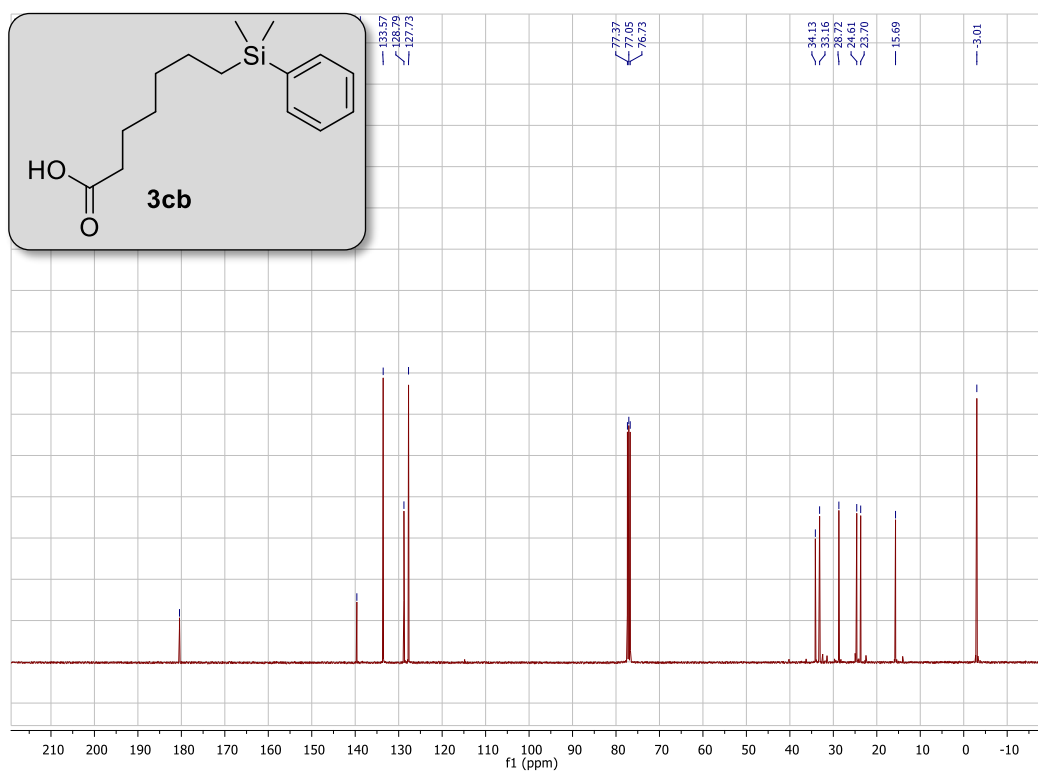
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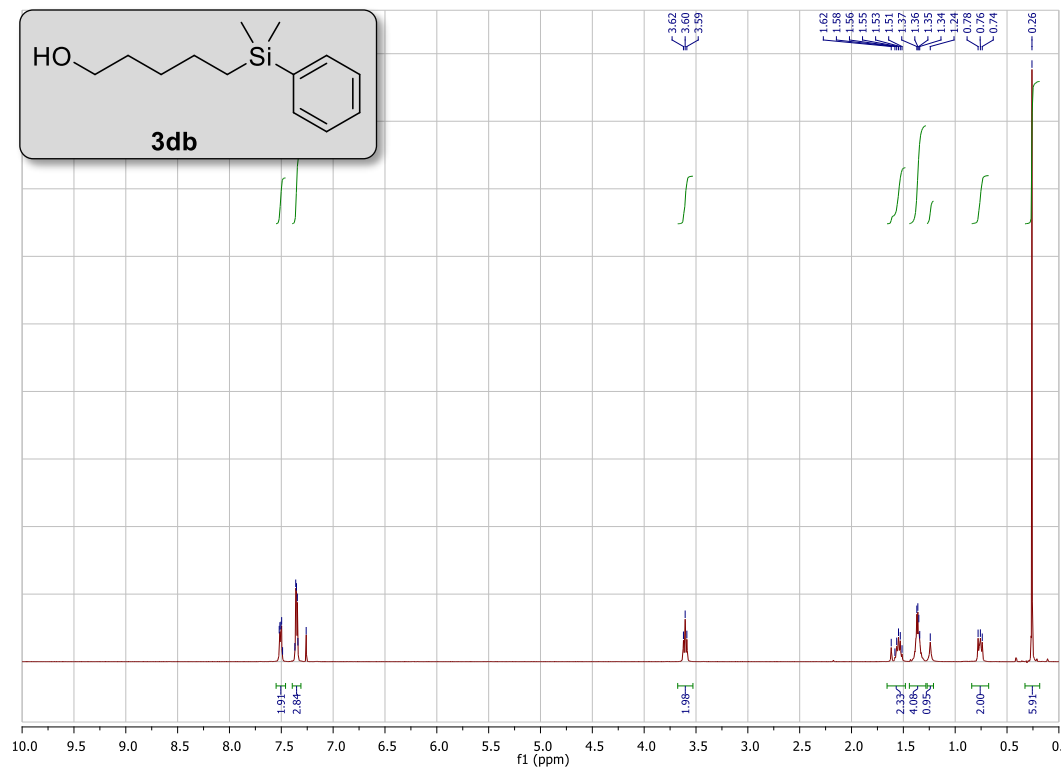
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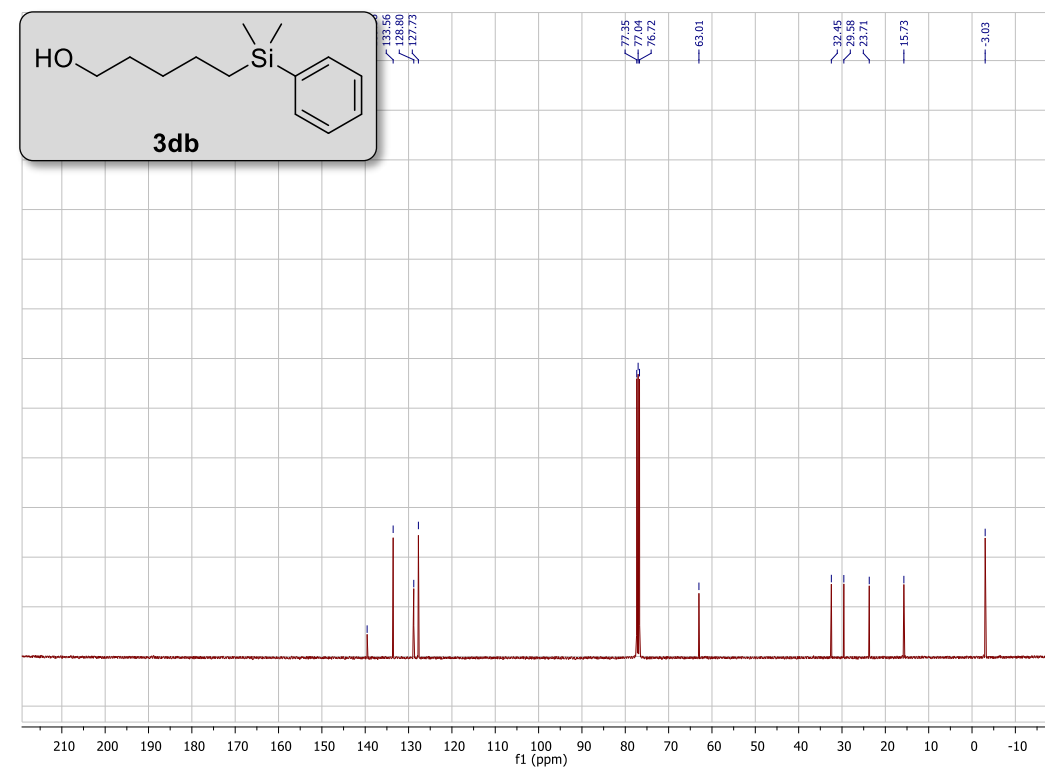
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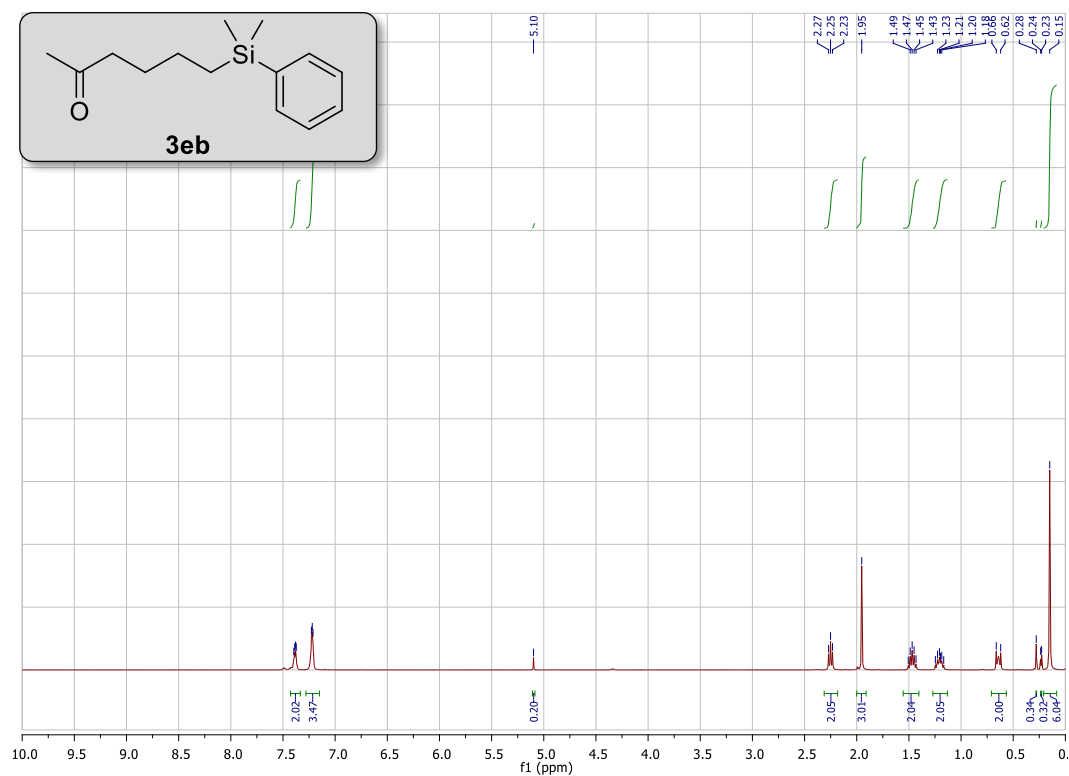
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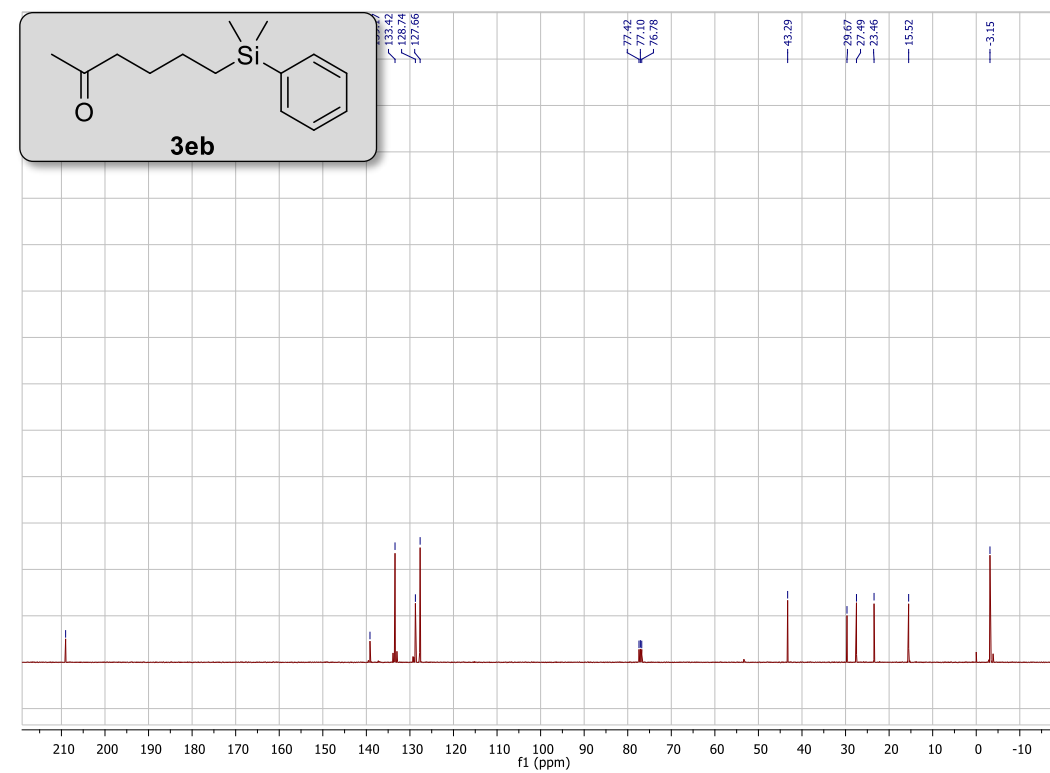
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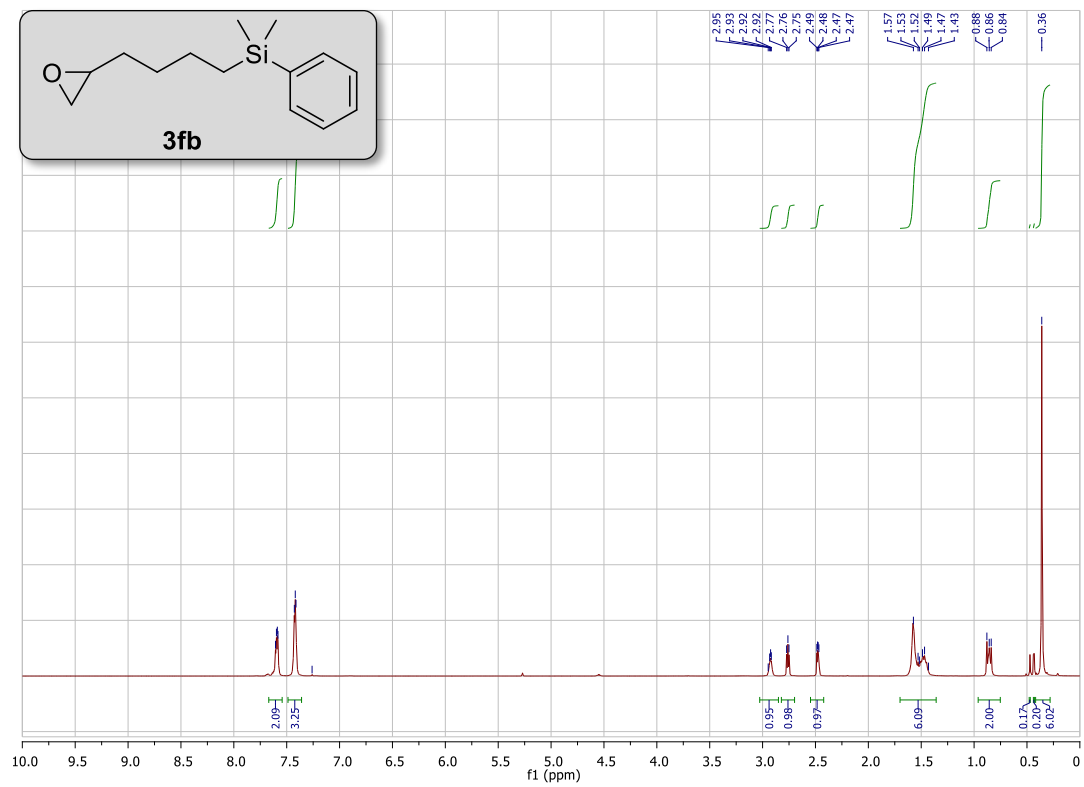
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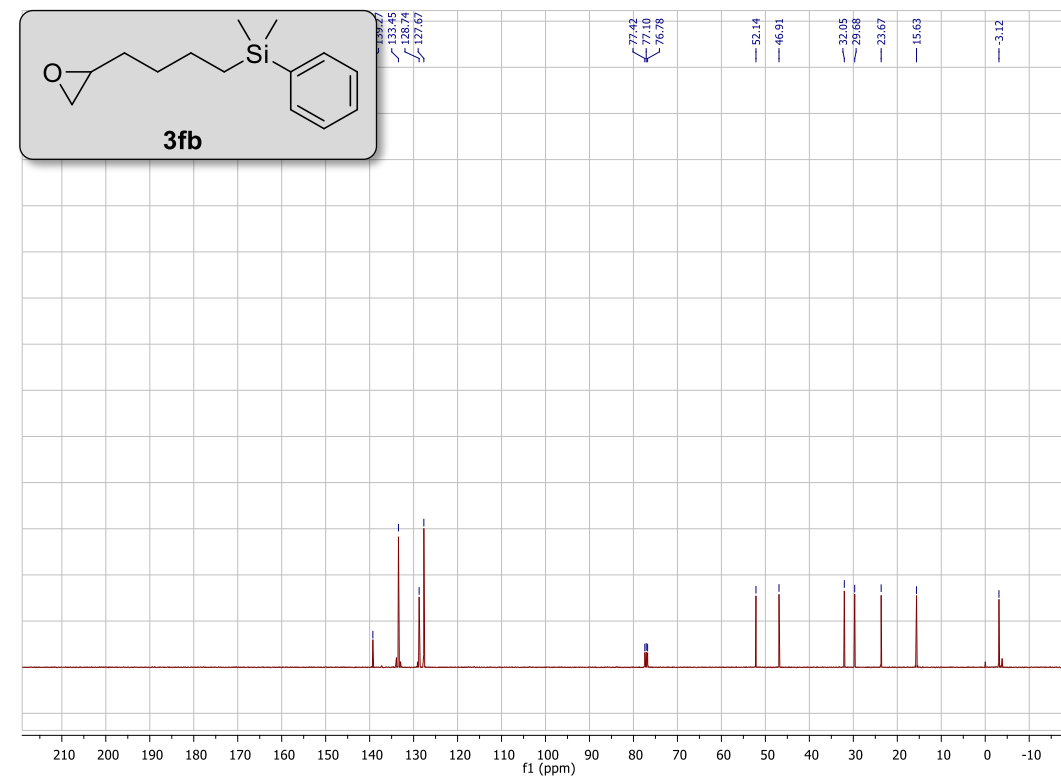
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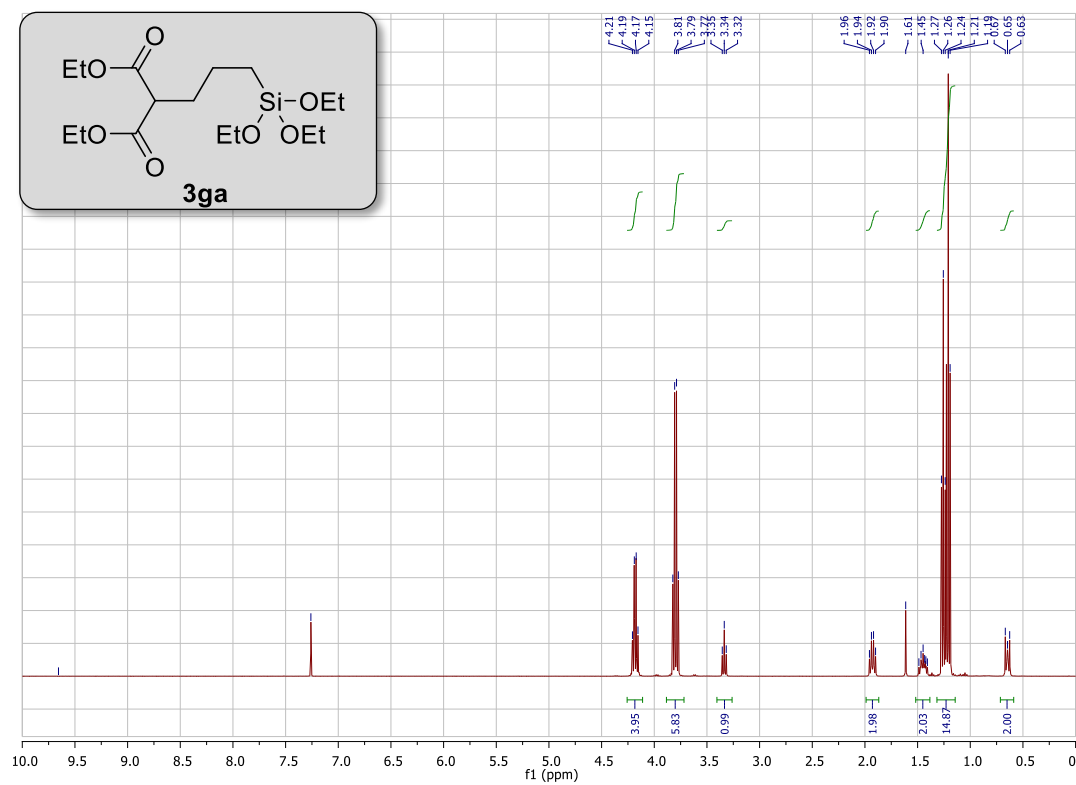
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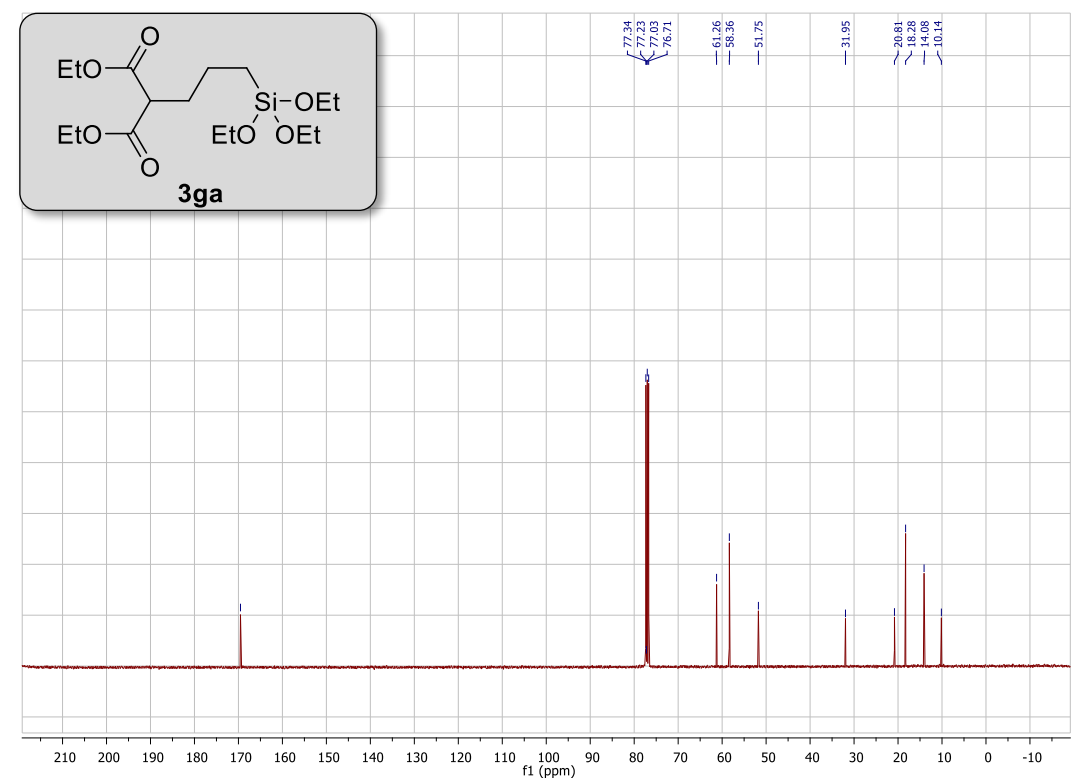
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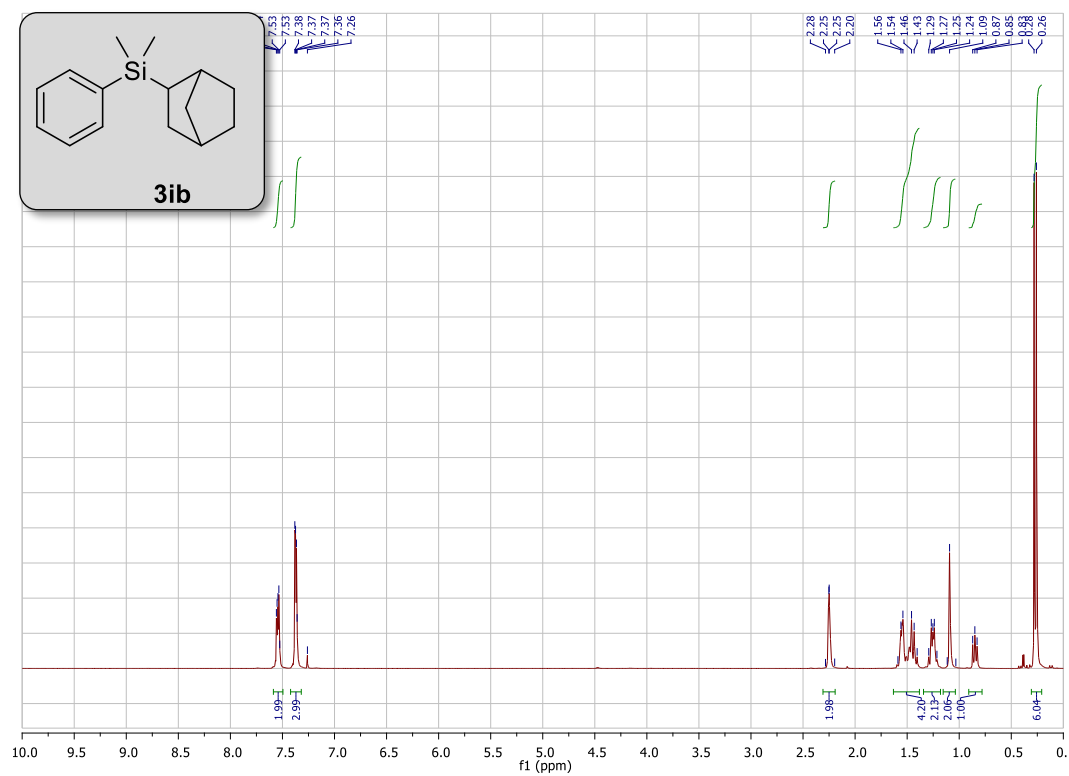
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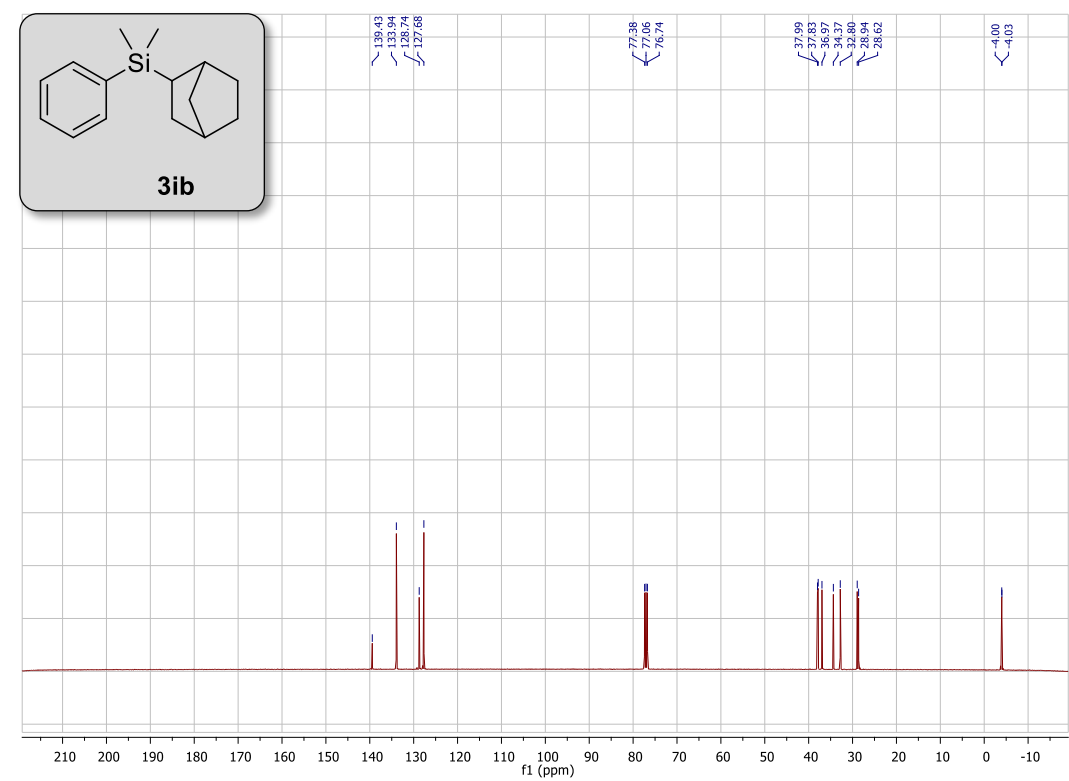
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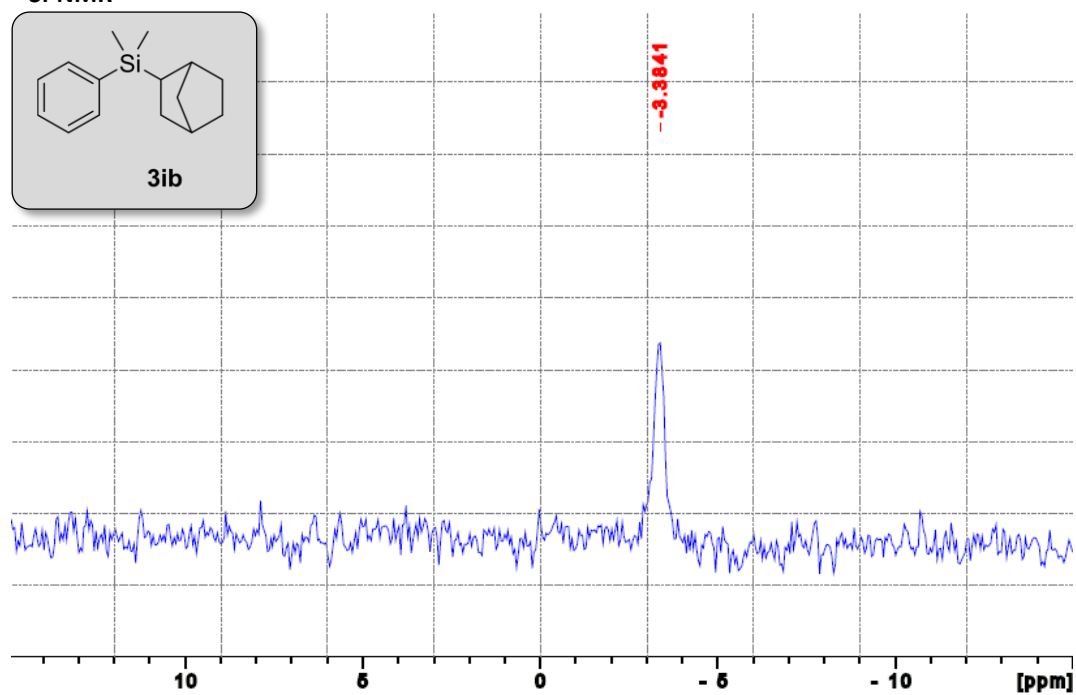
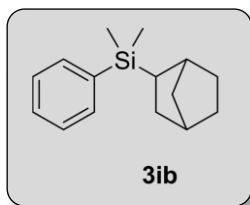
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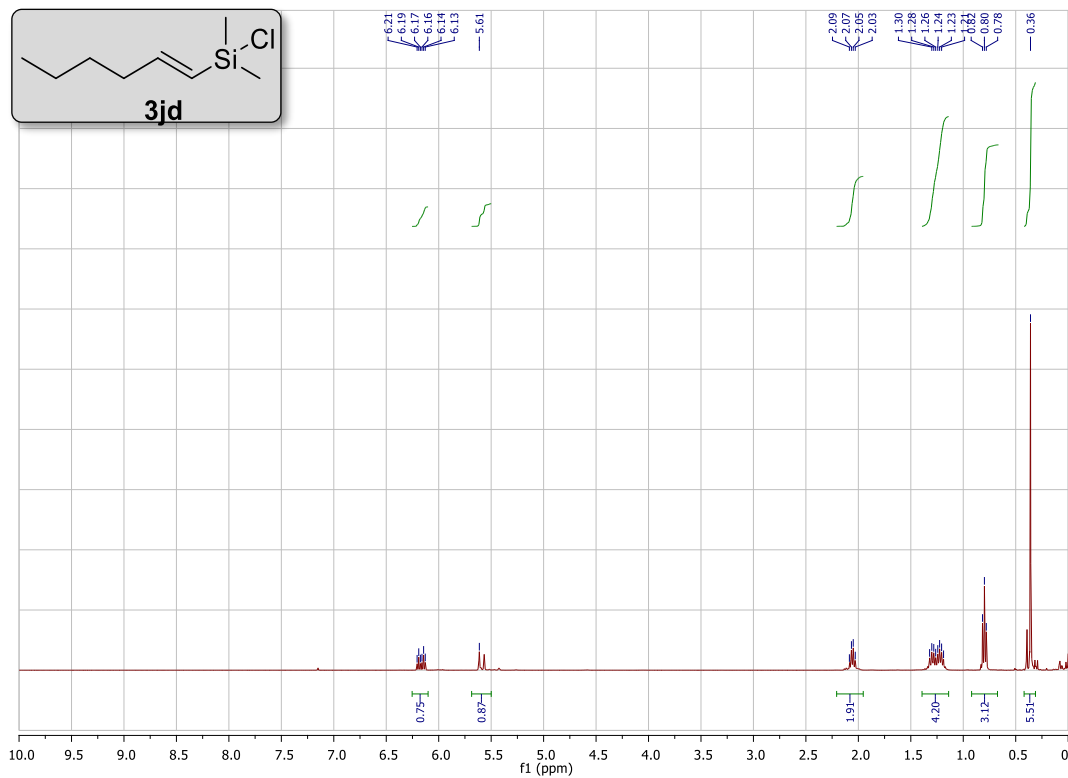
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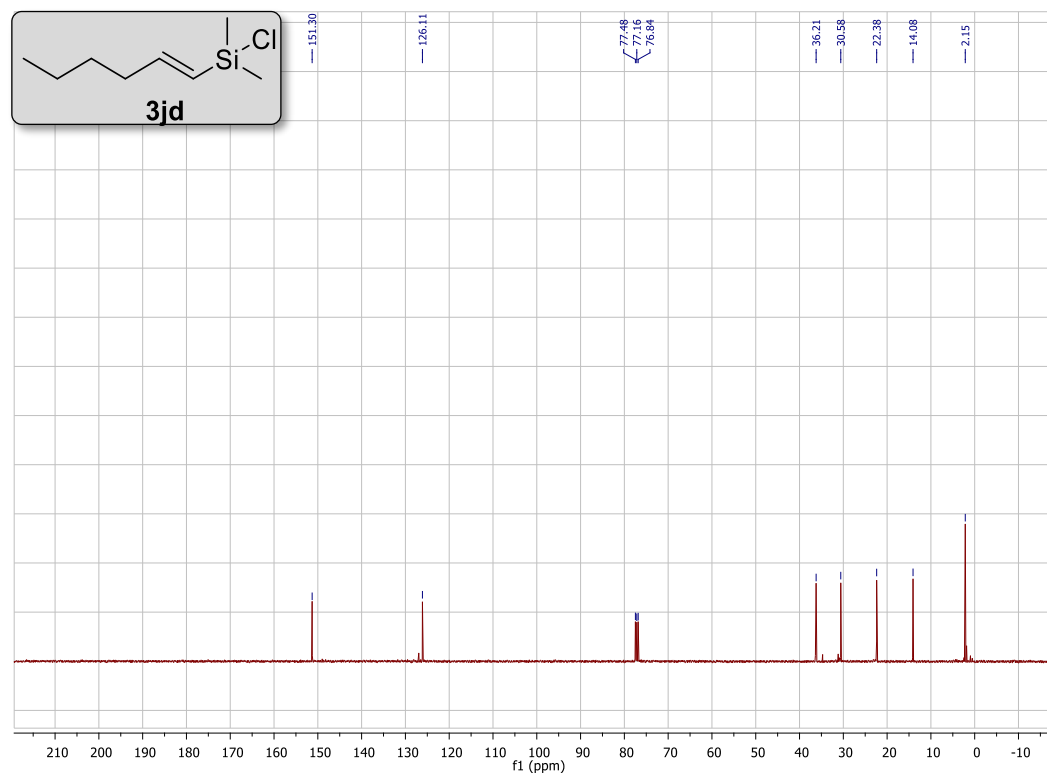
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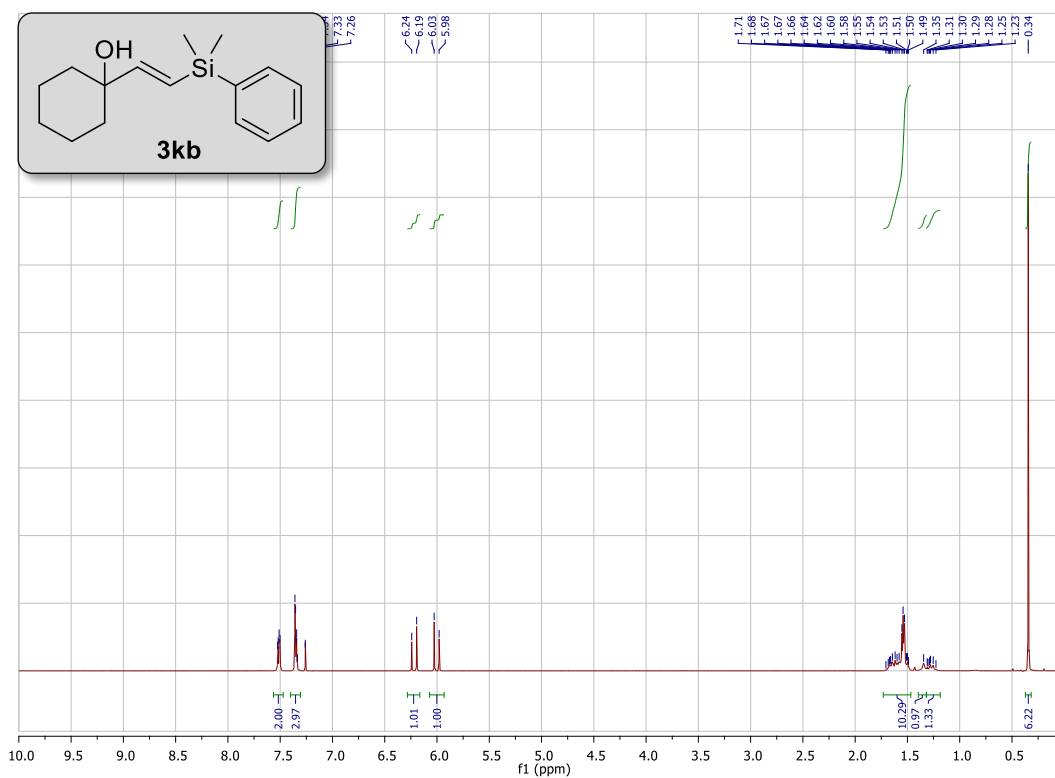
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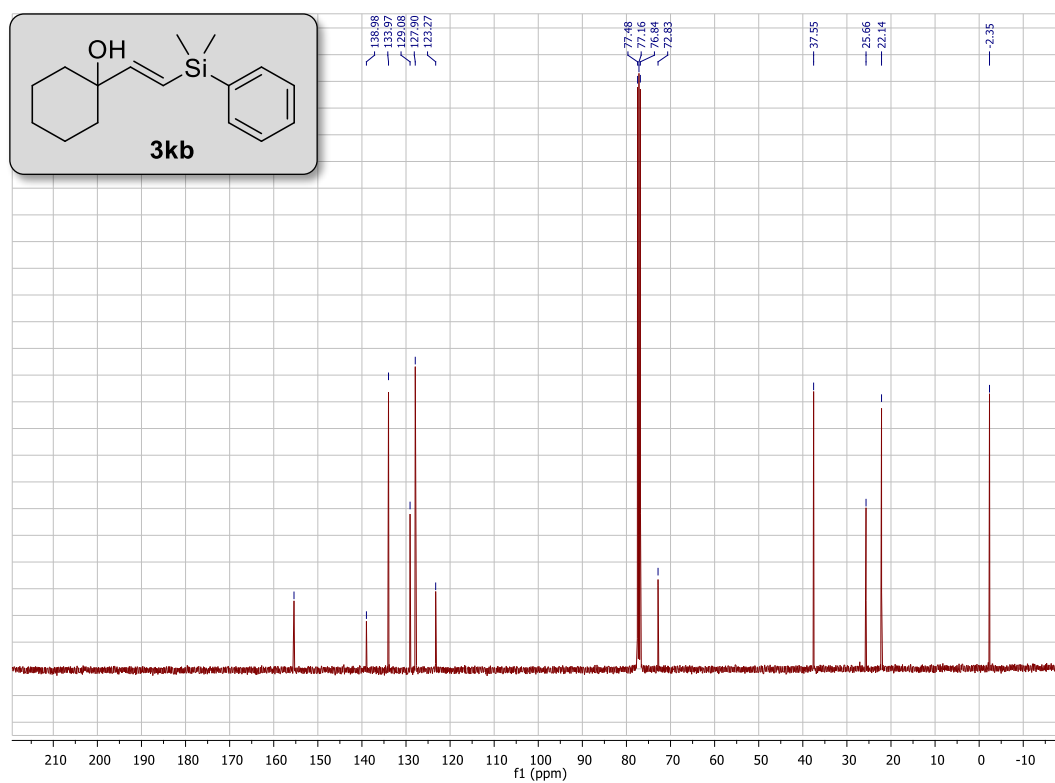
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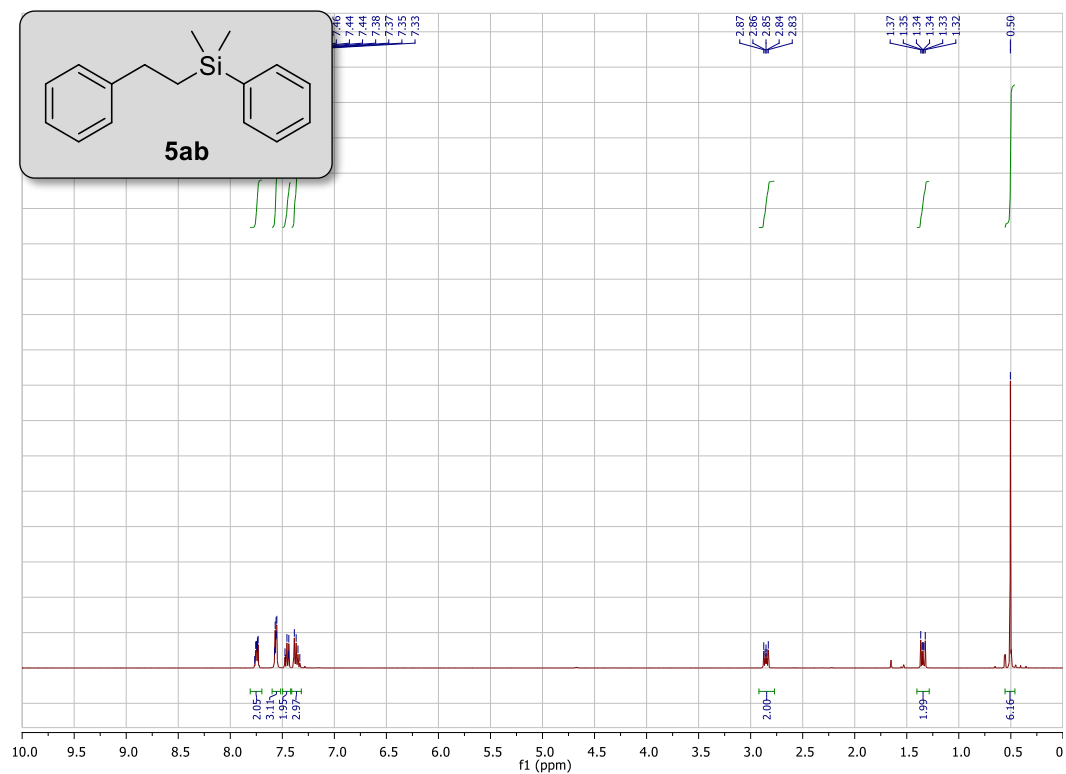
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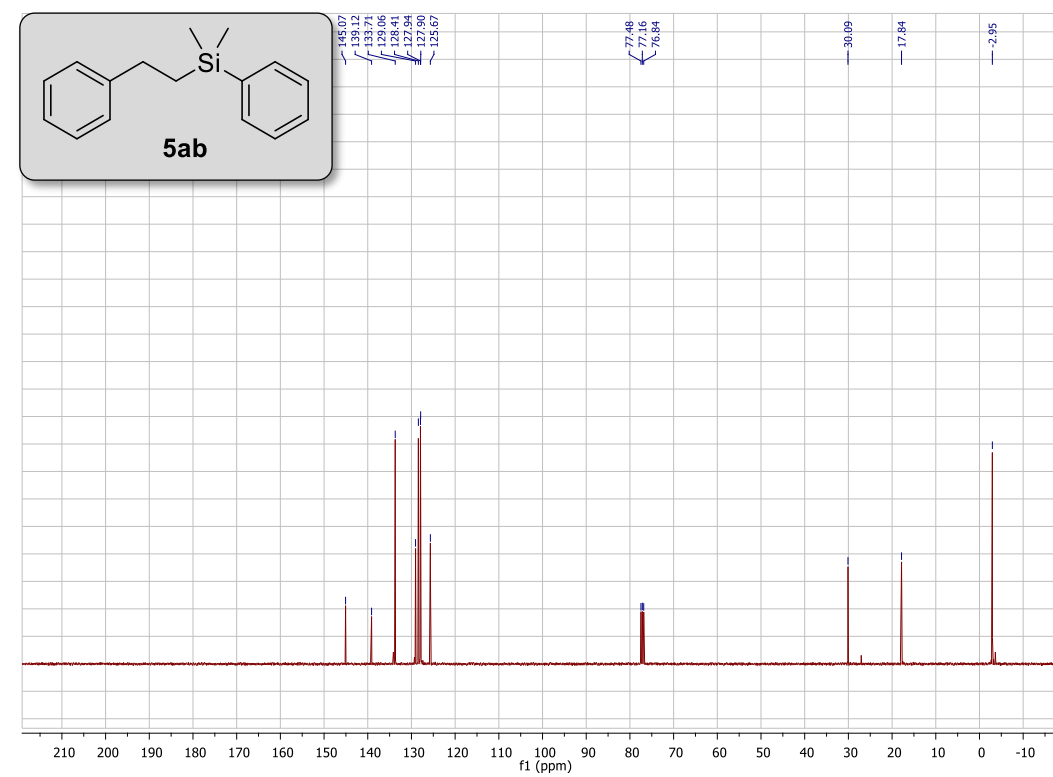
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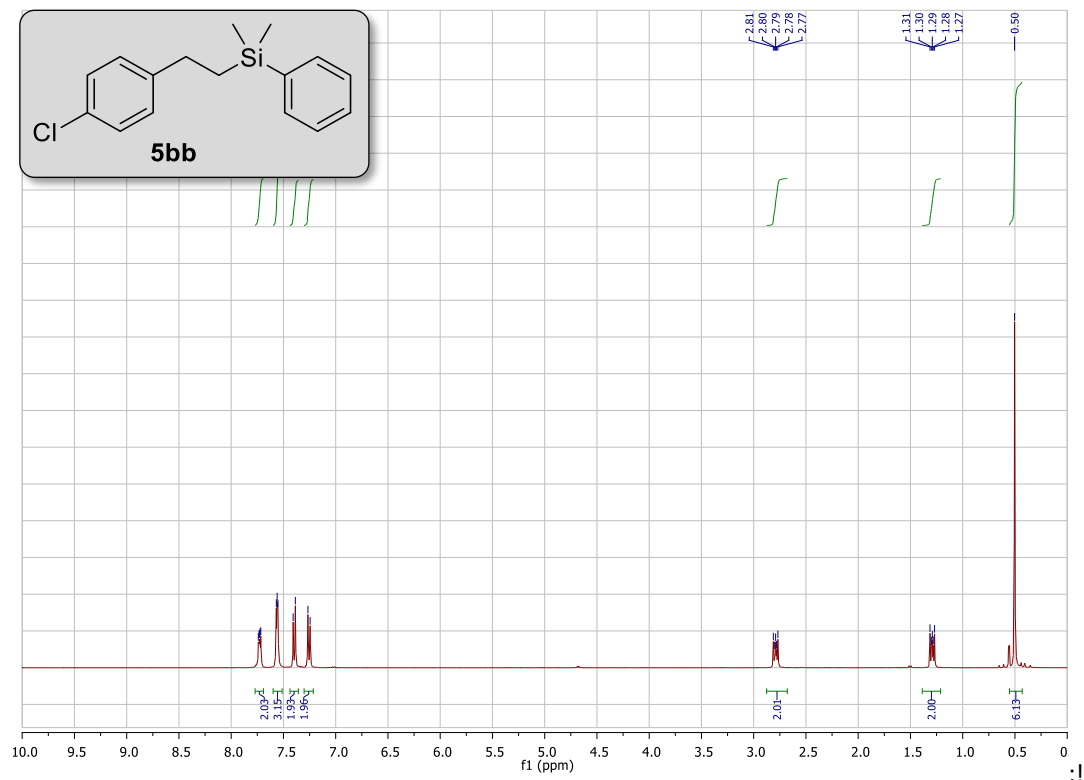
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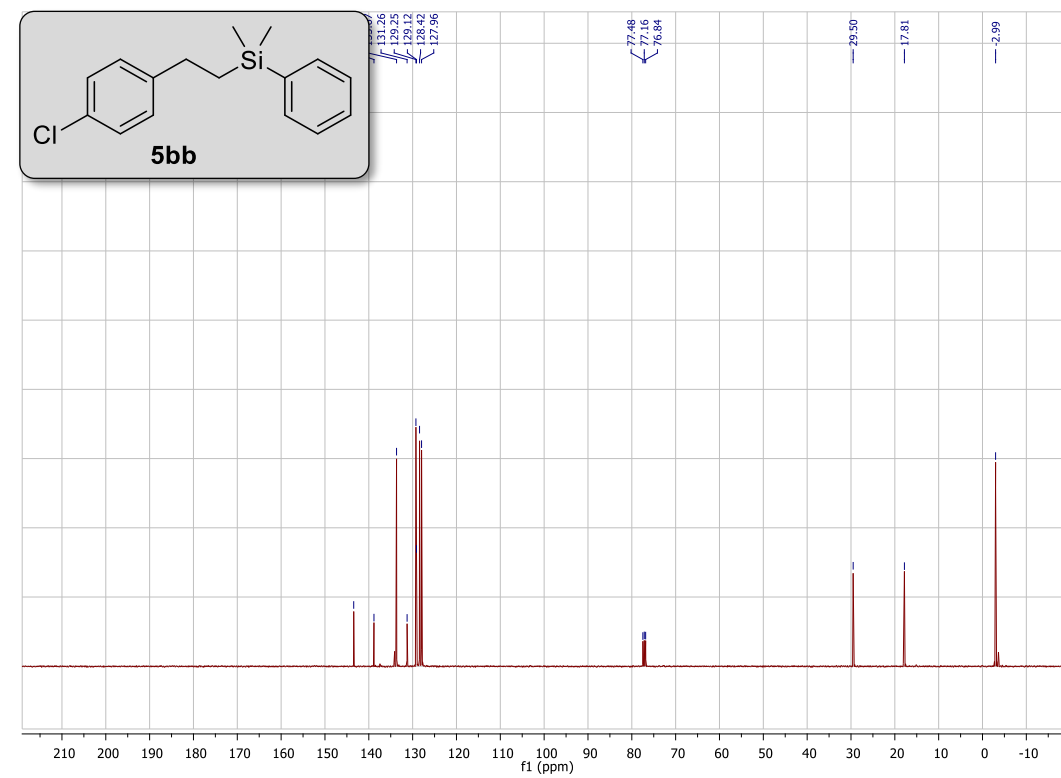
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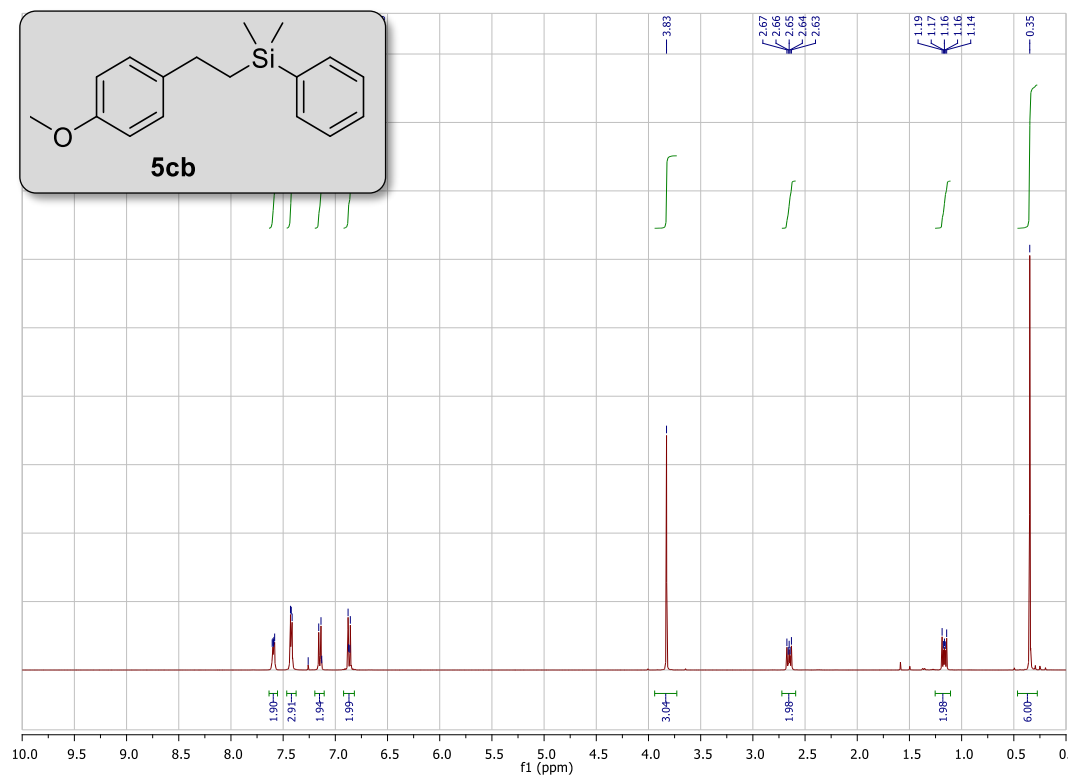
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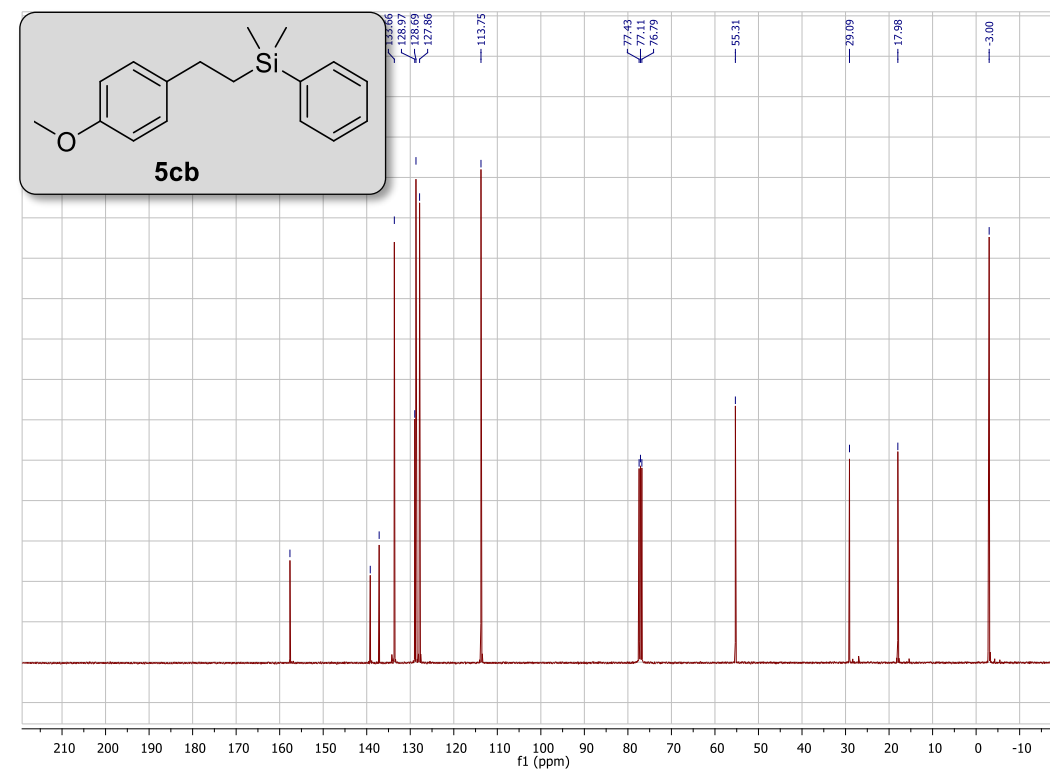
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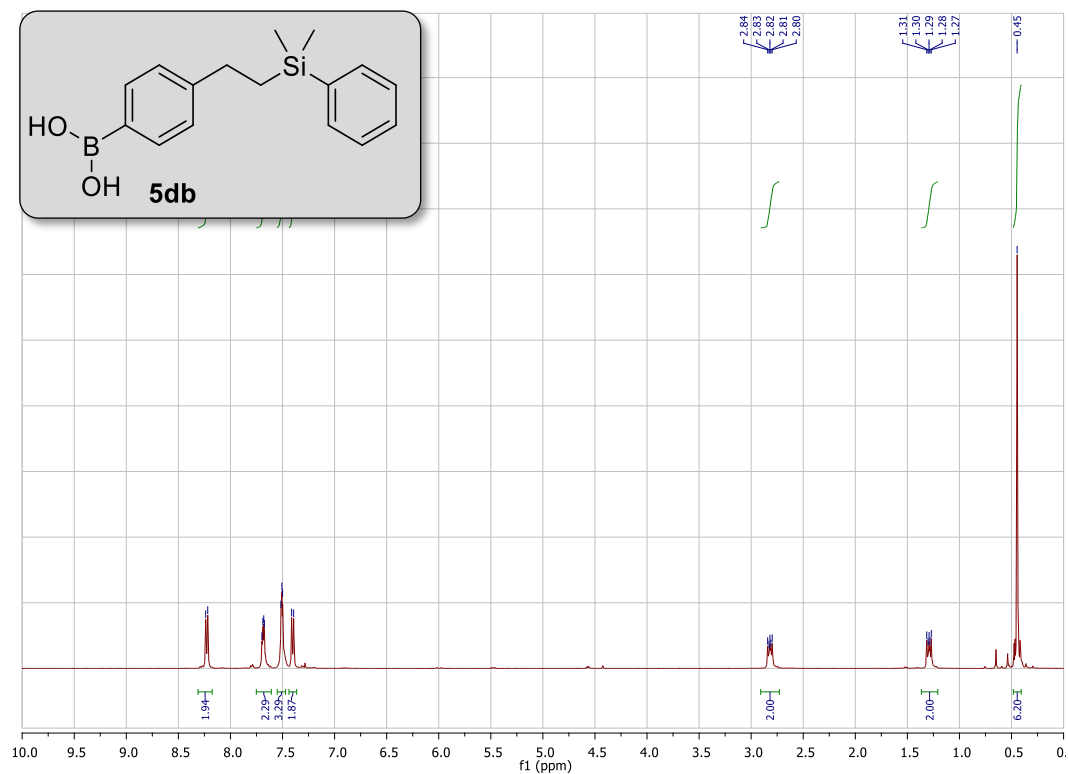
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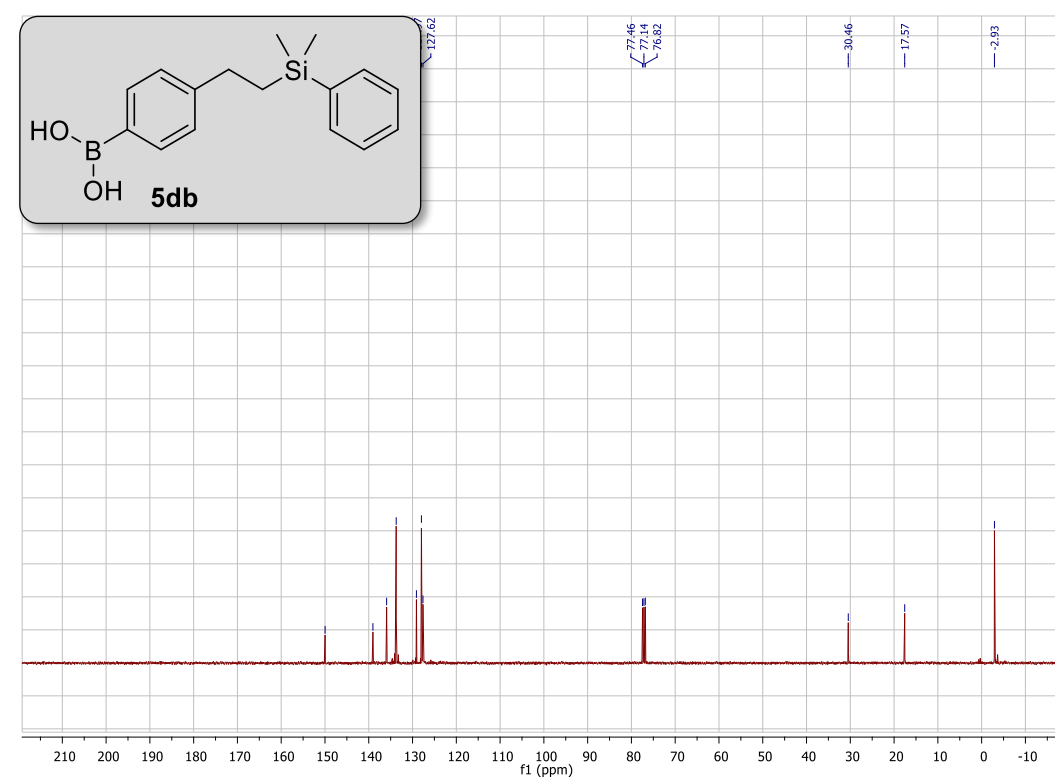
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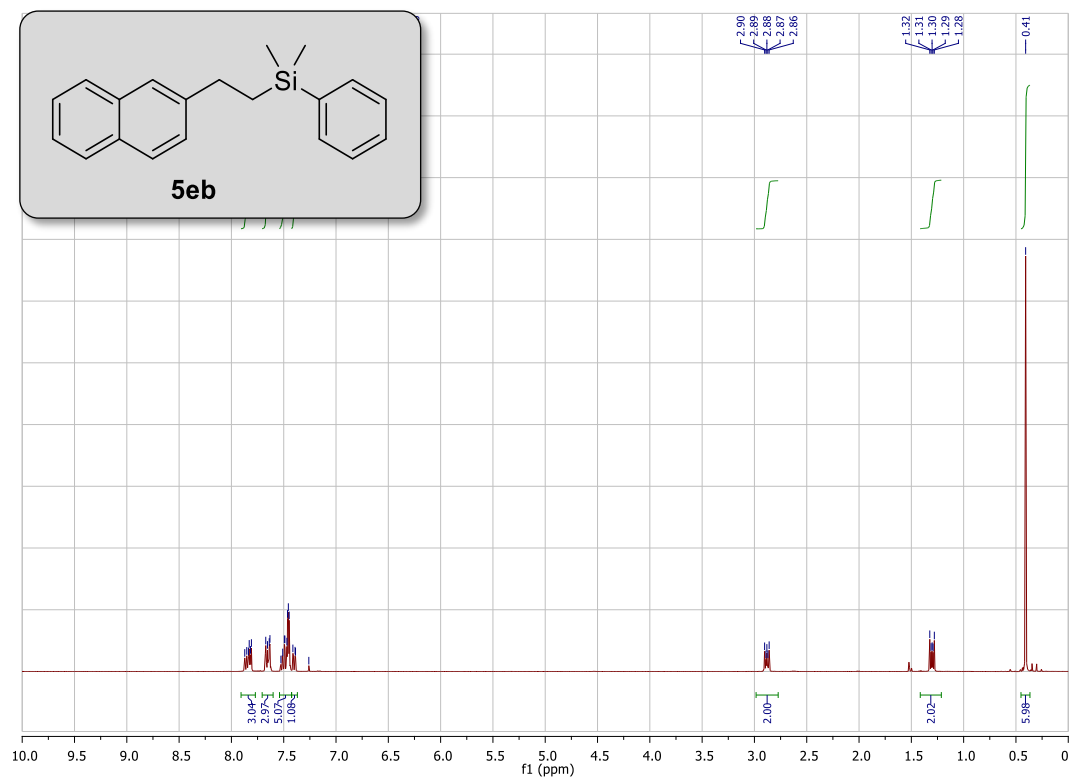
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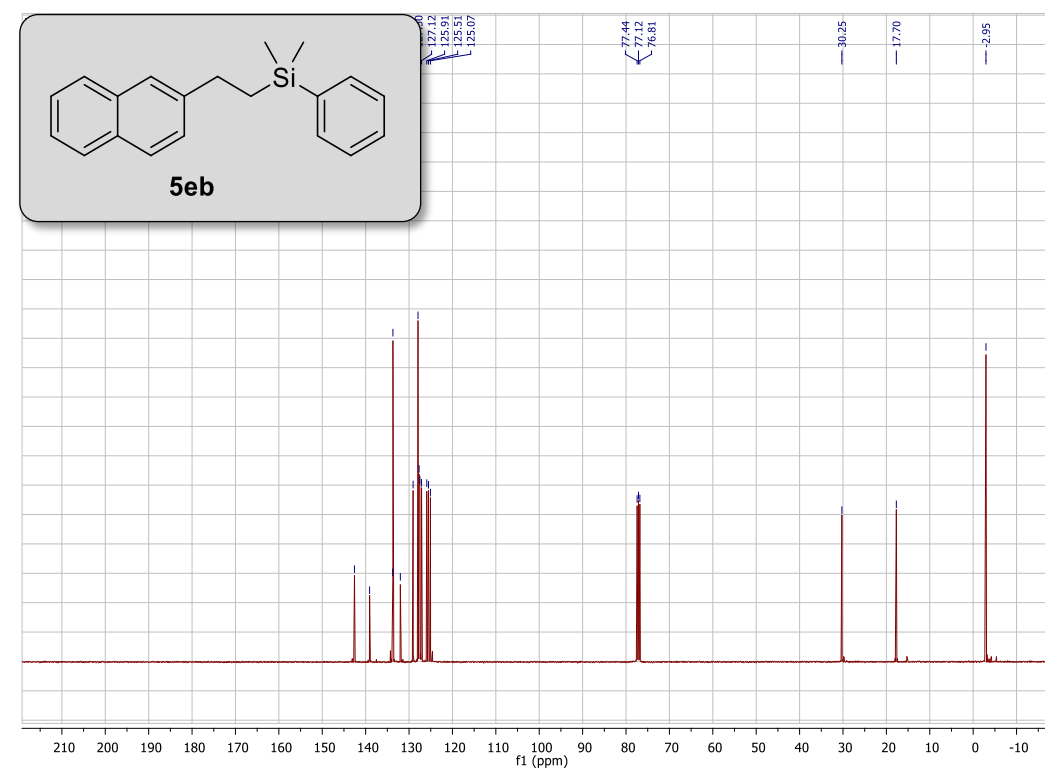
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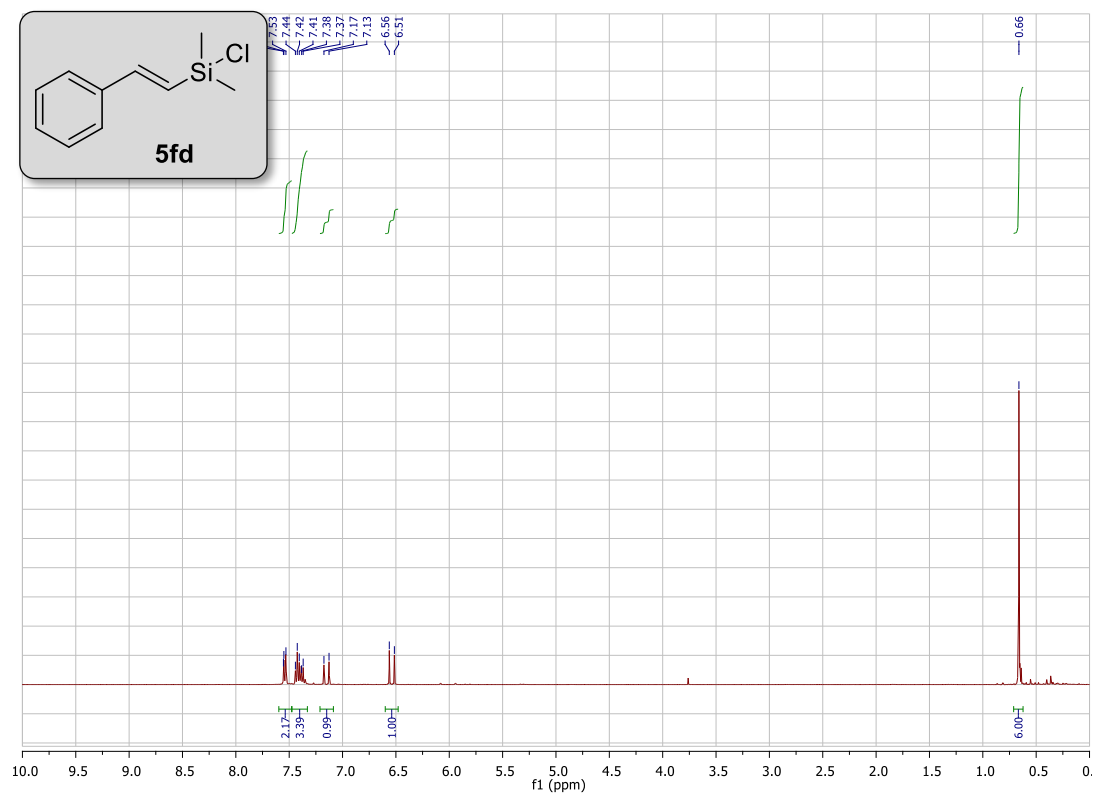
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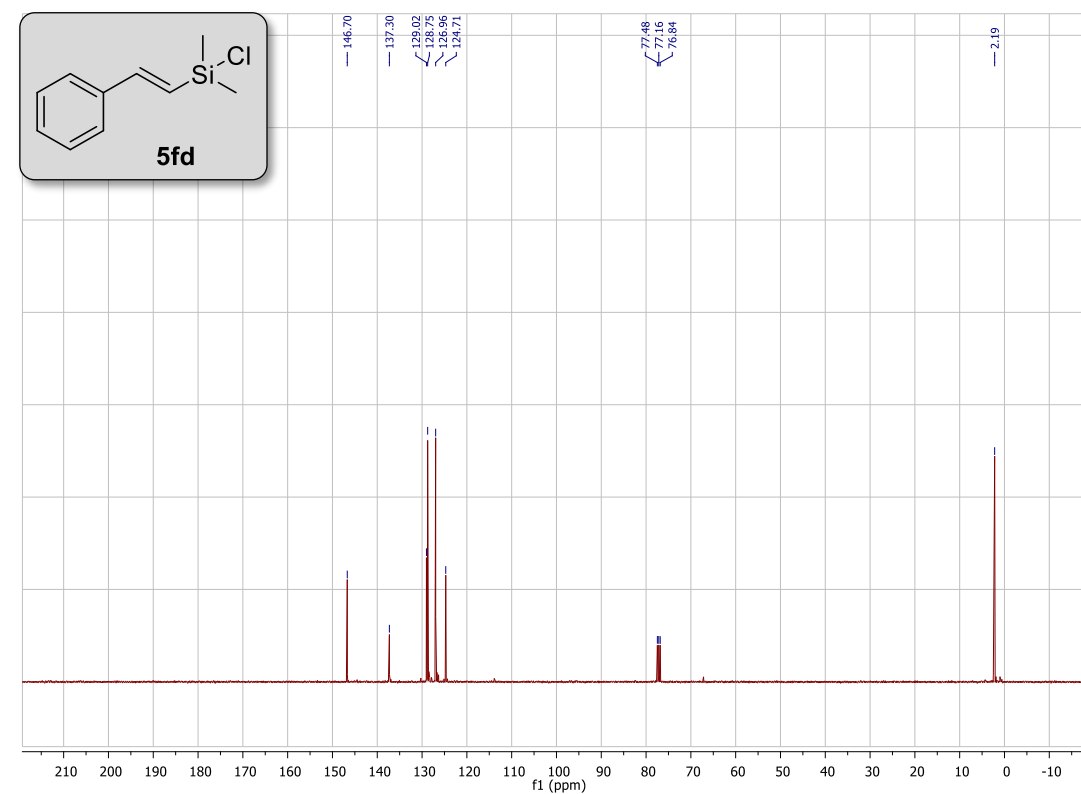
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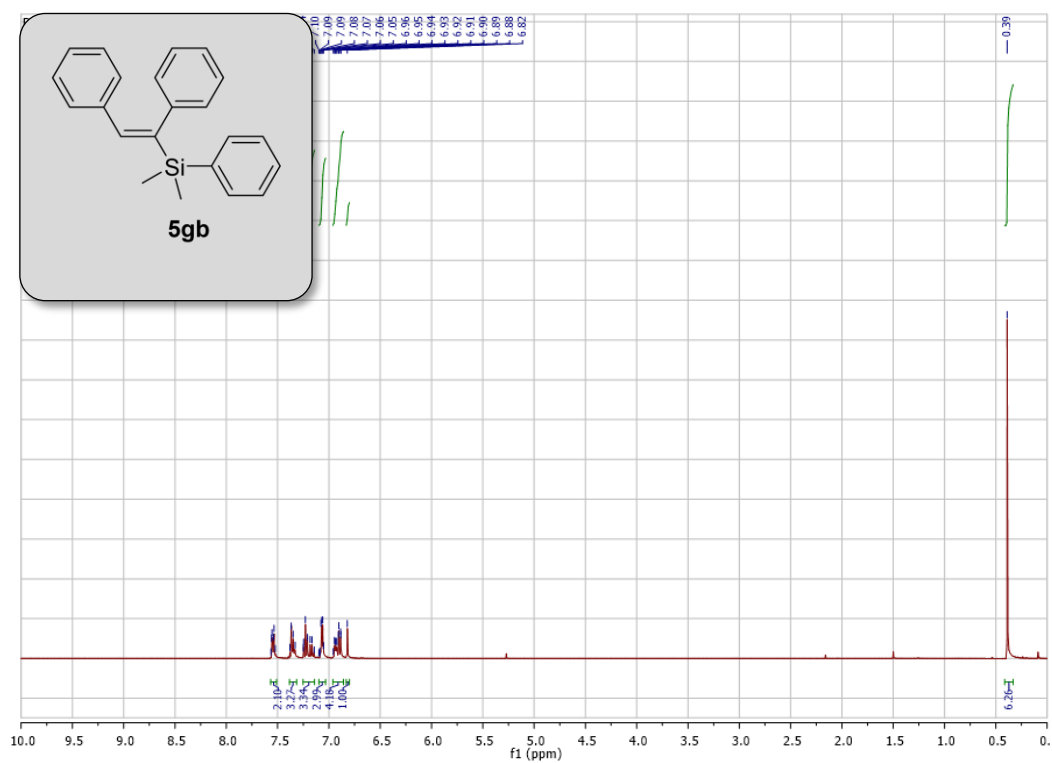
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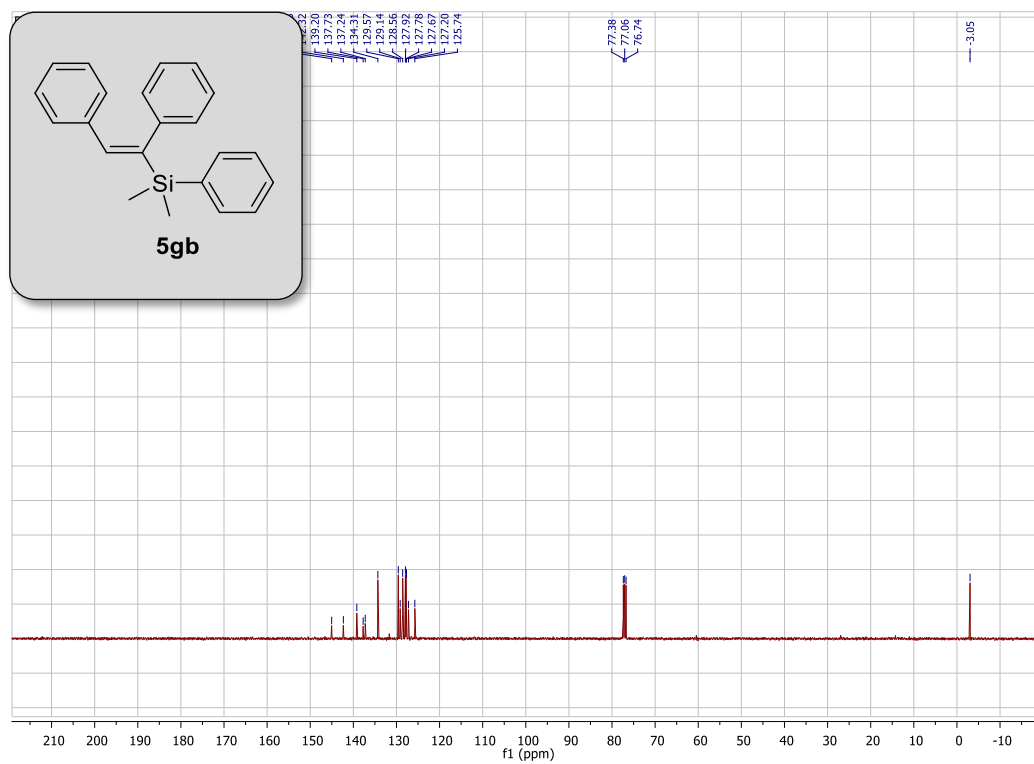
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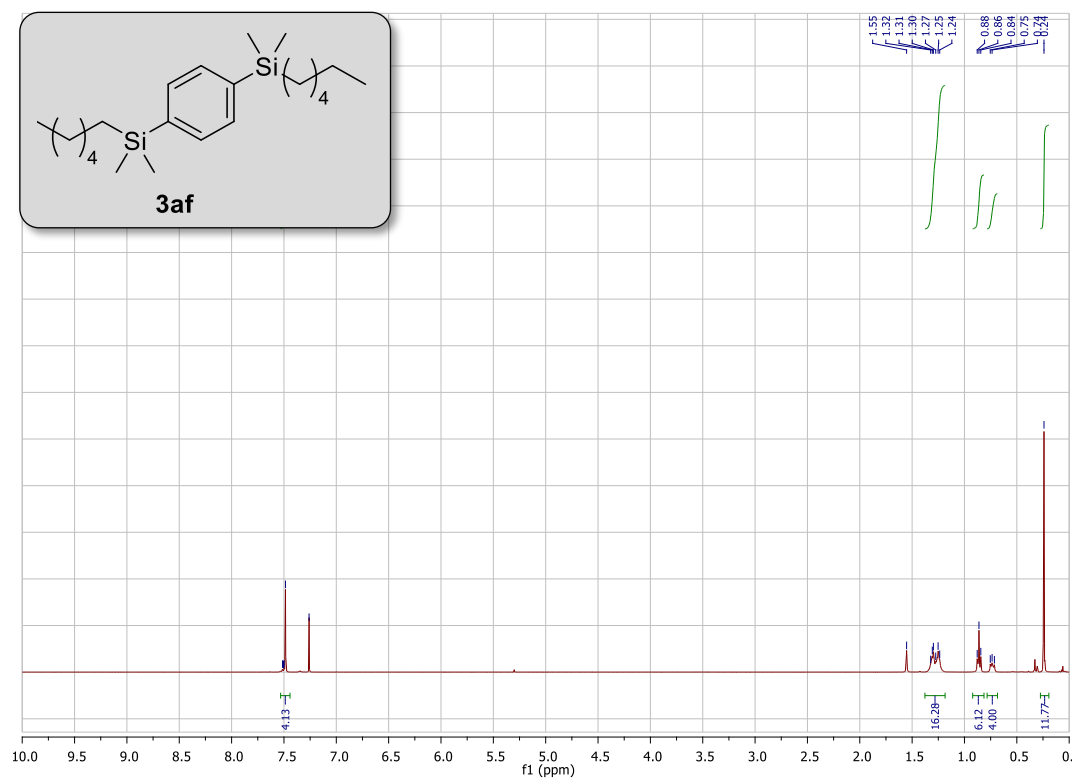
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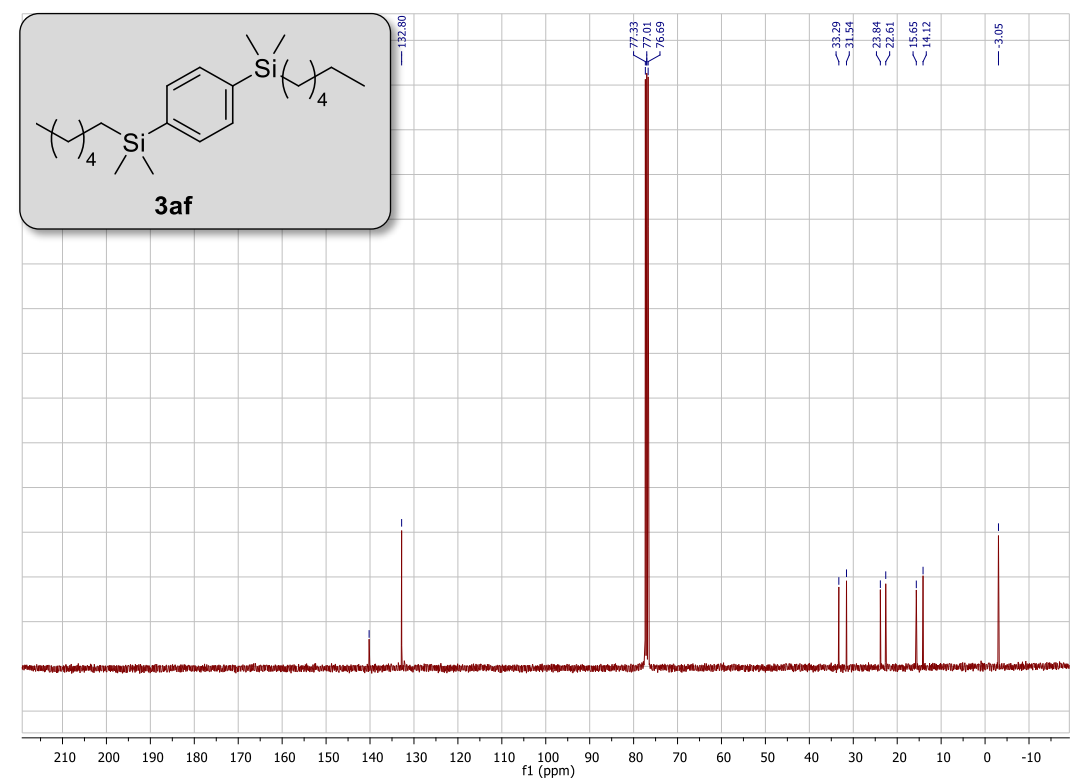
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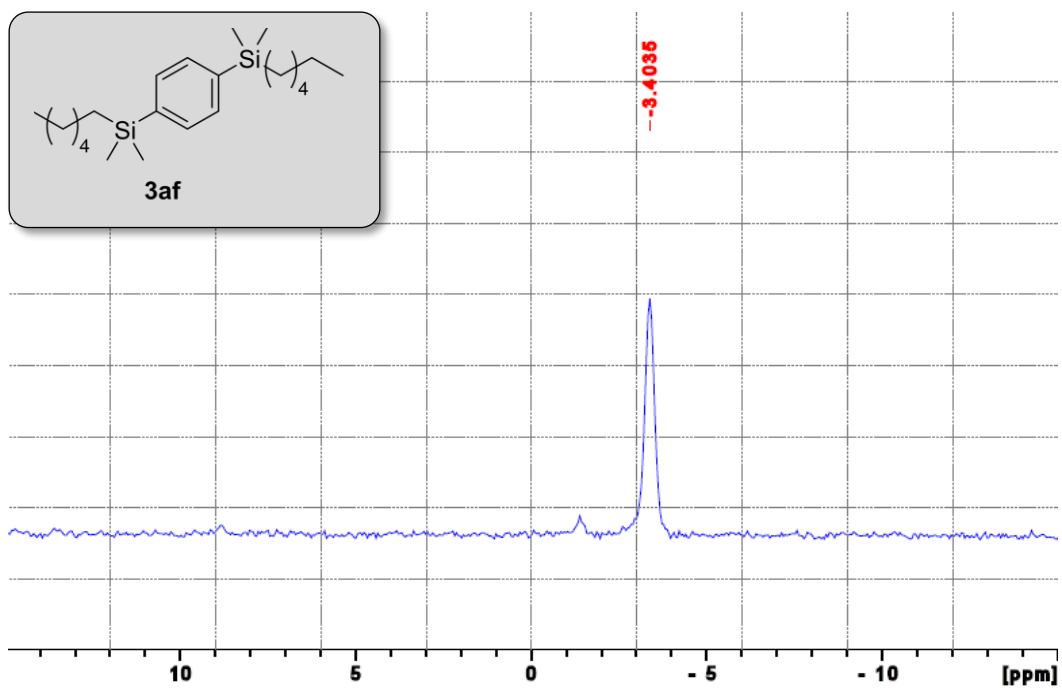
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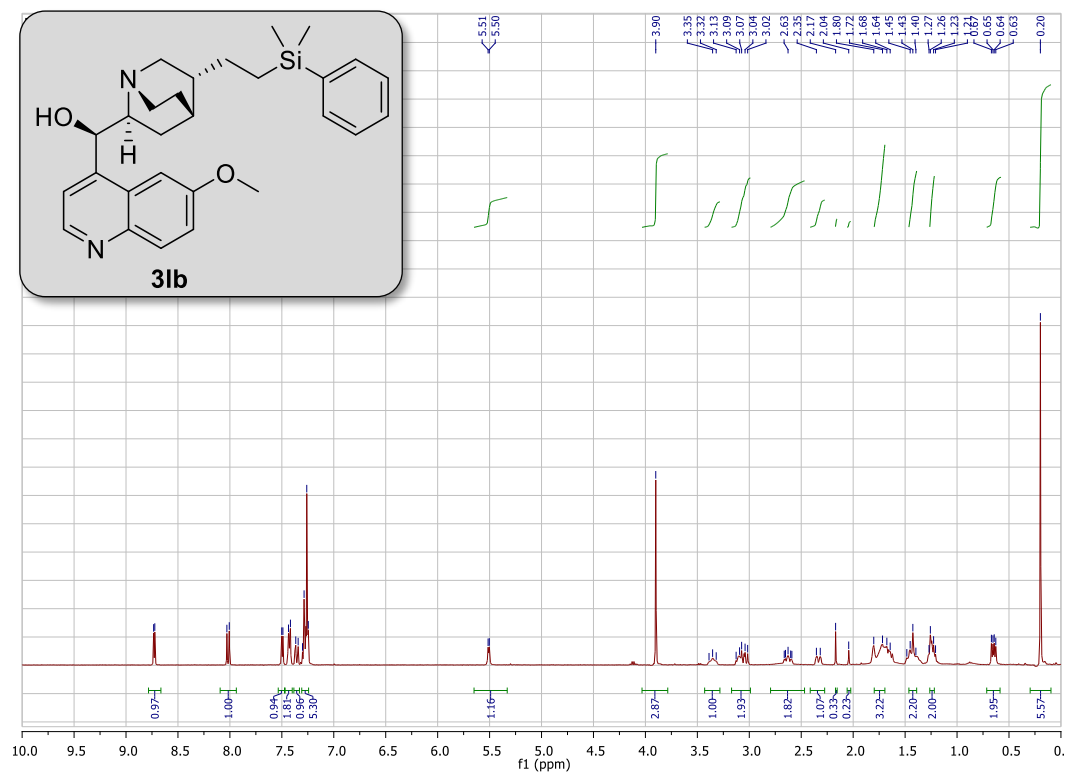
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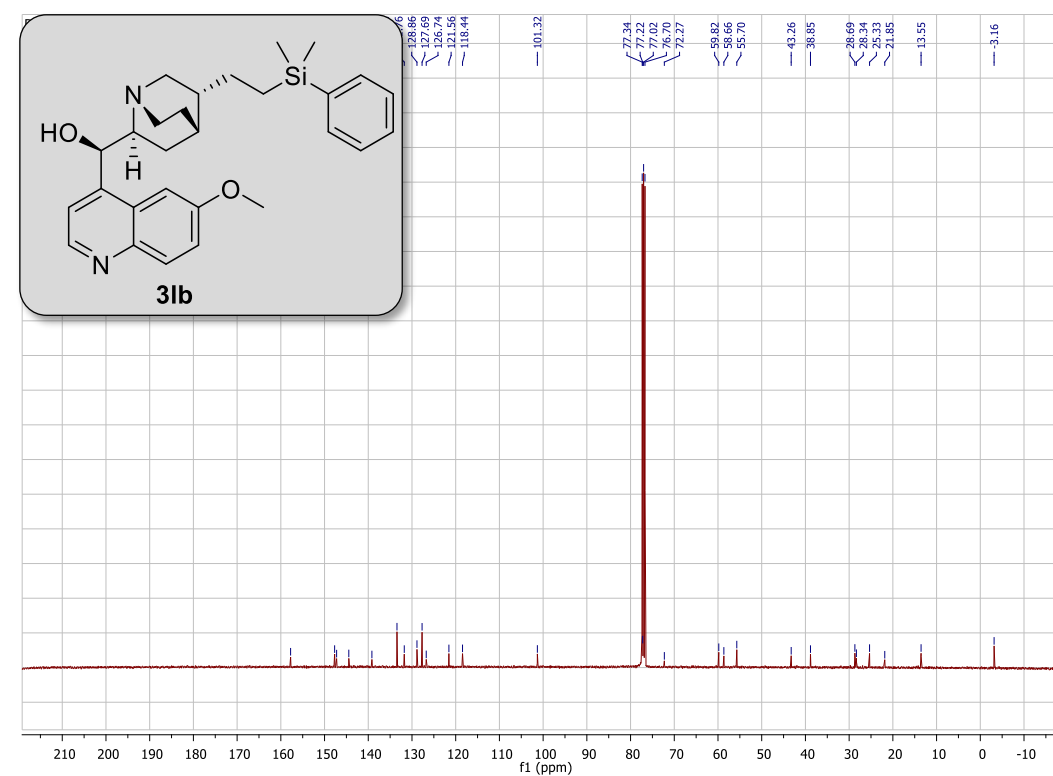
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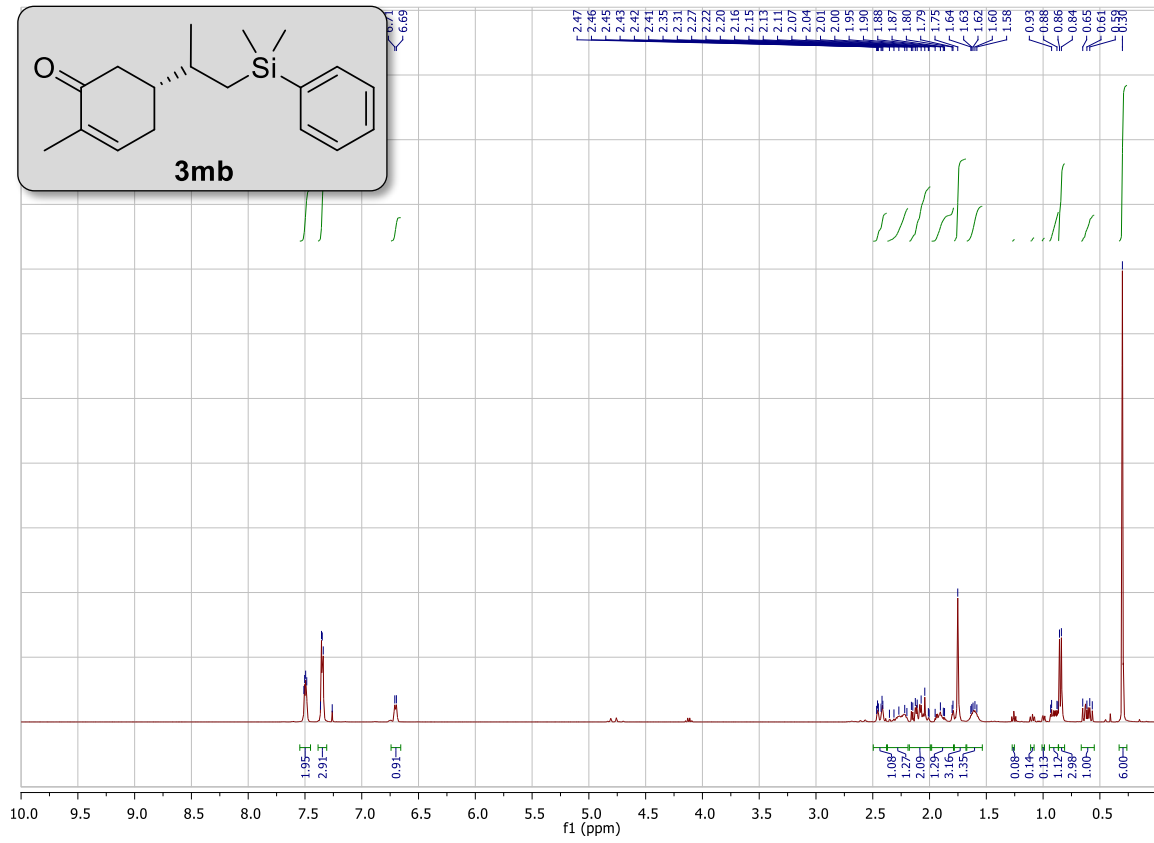
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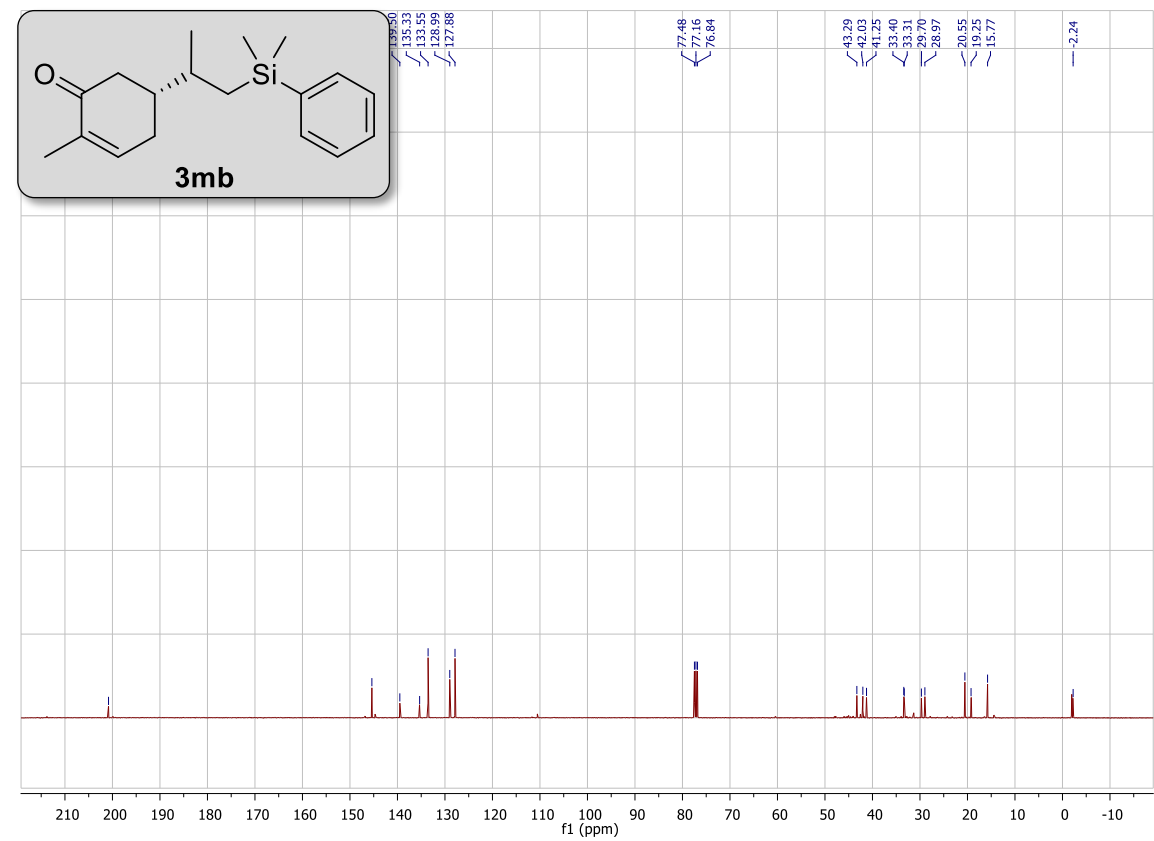
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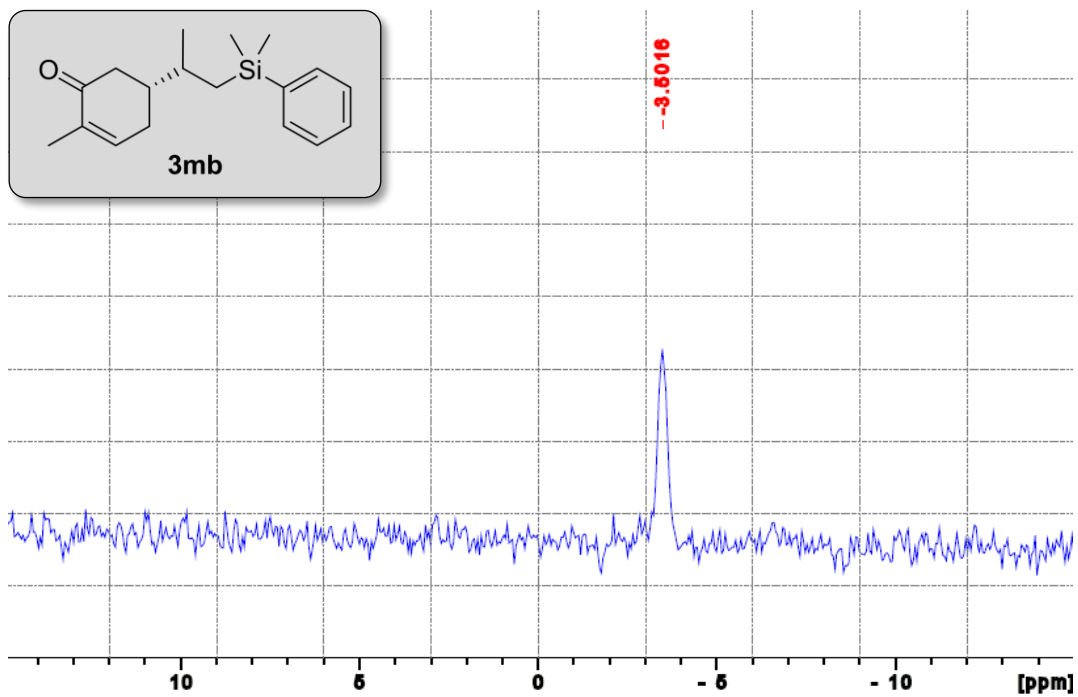
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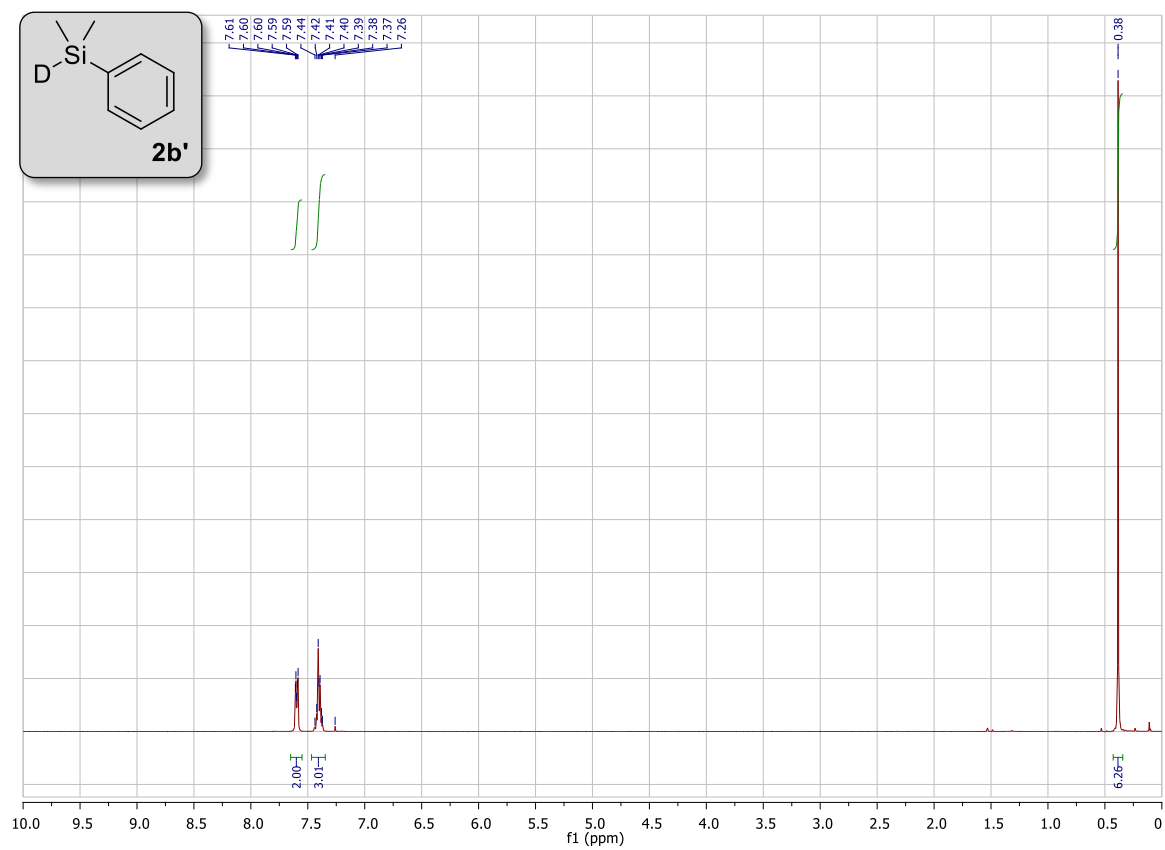
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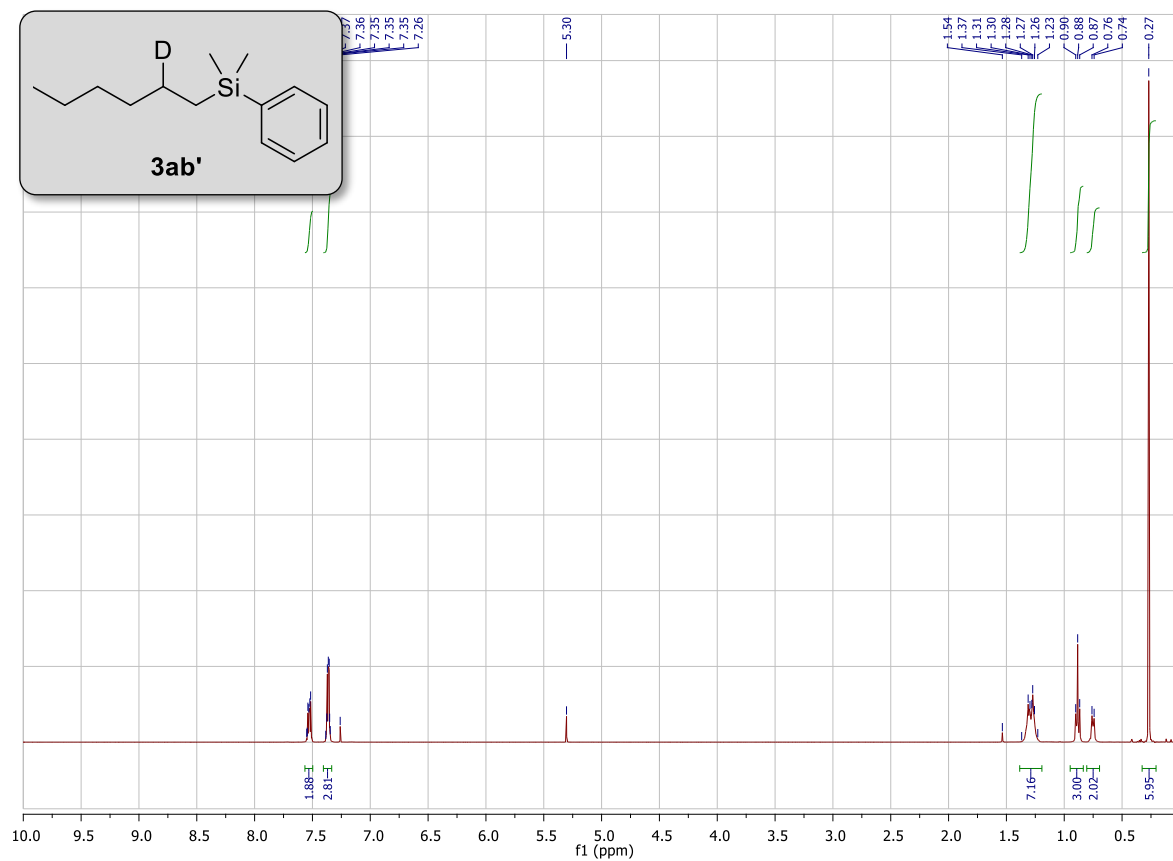
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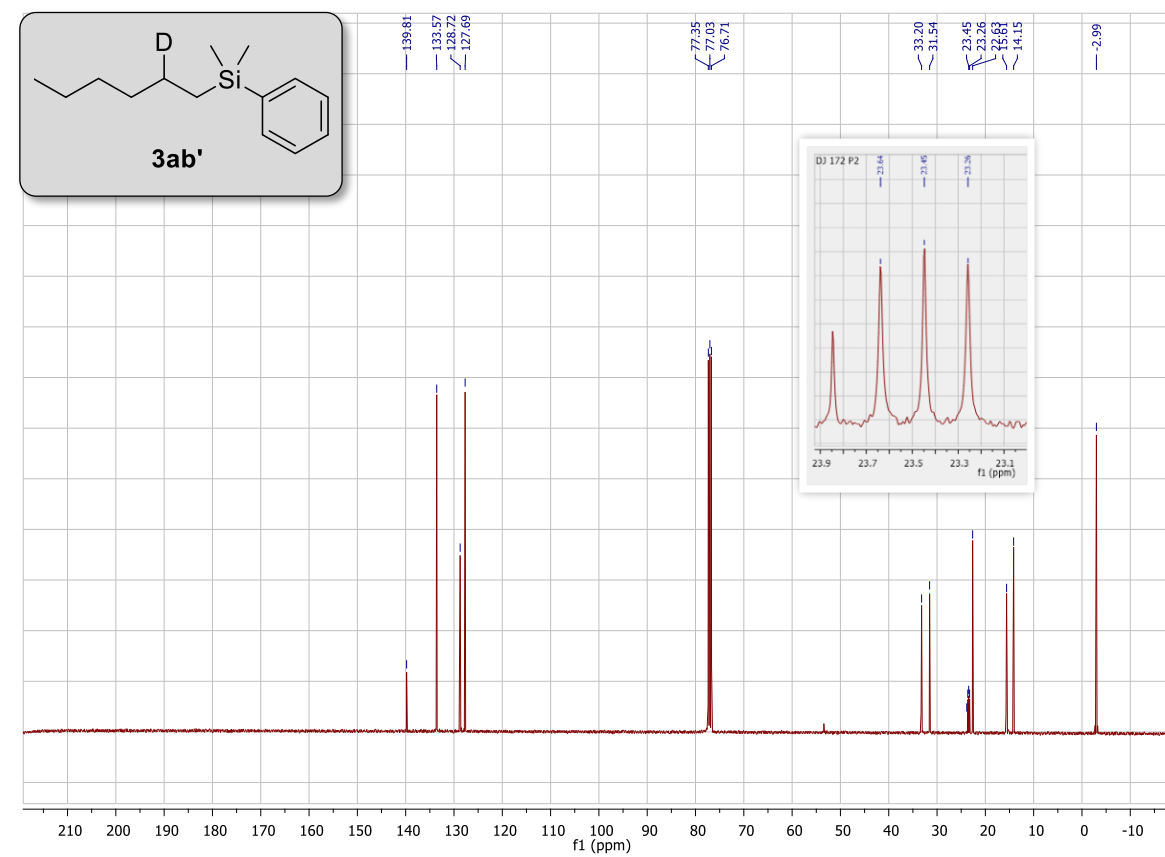
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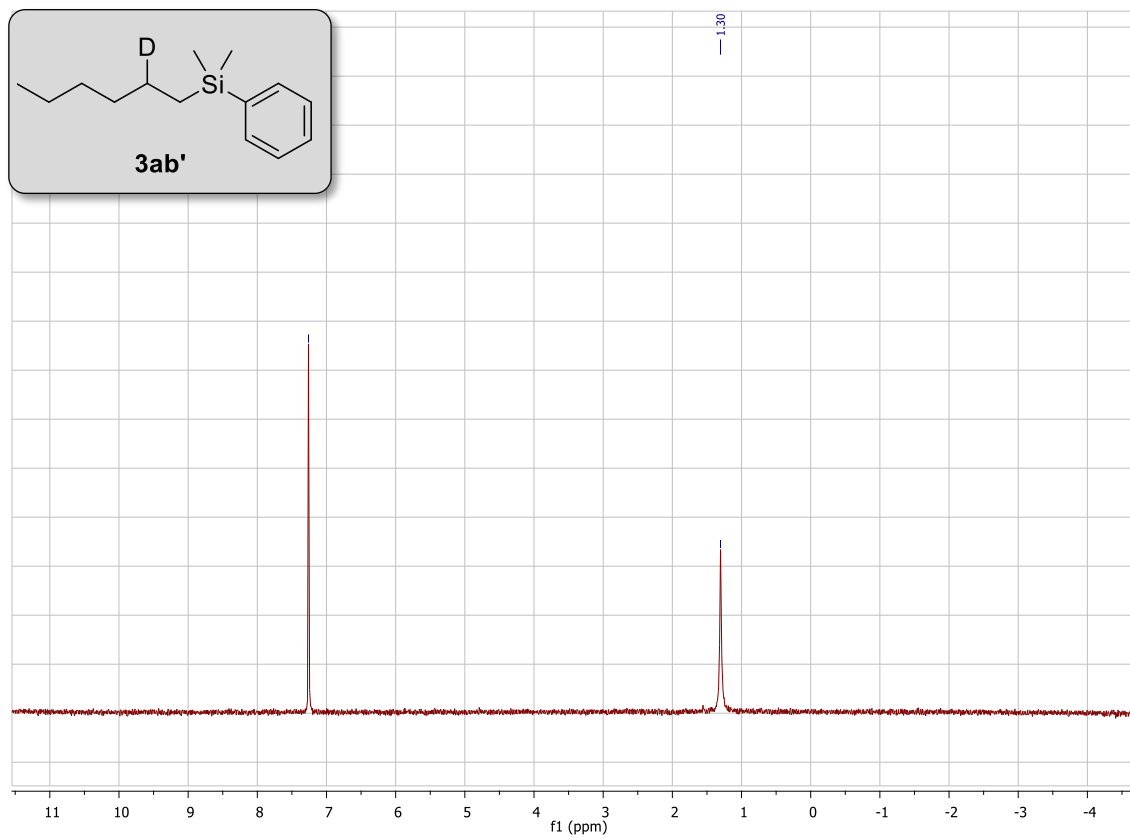
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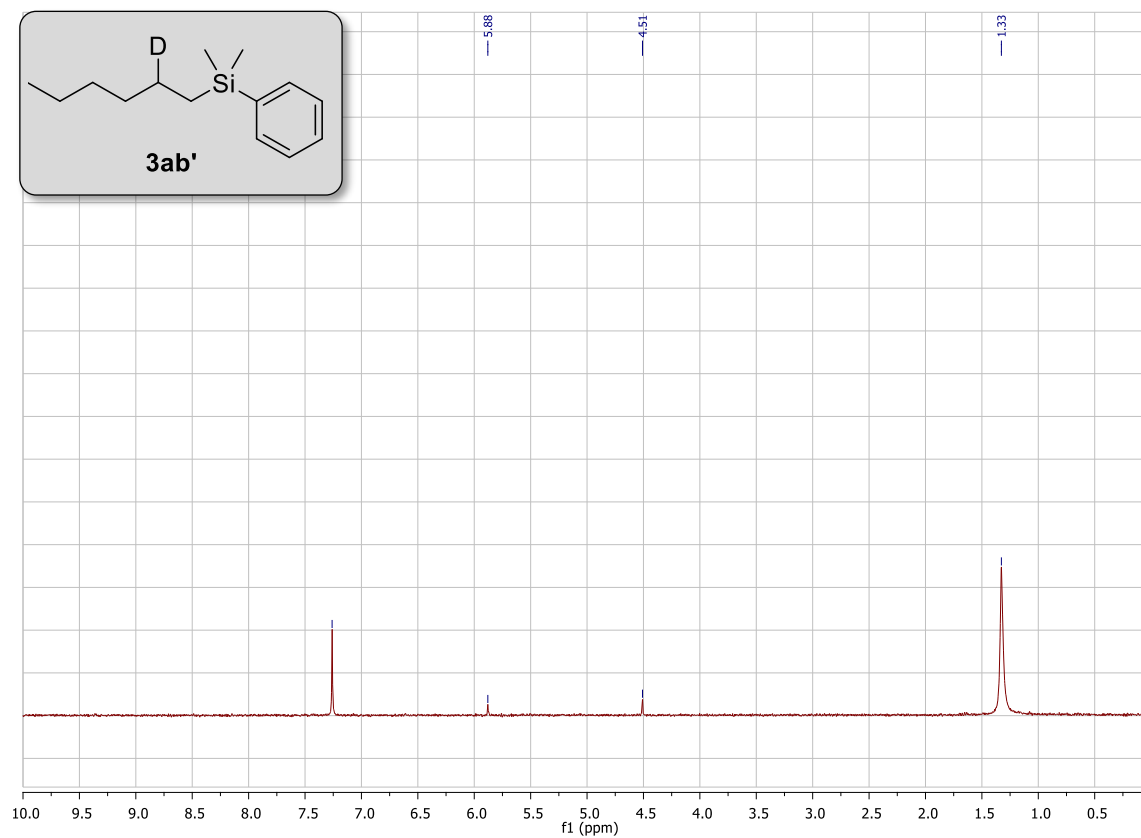
¹³C-NMR



$^2\text{H-NMR}$



$^2\text{H-NMR}$ of the crude reaction mixture after 7 days (bottom).



3. Supplementary Figures

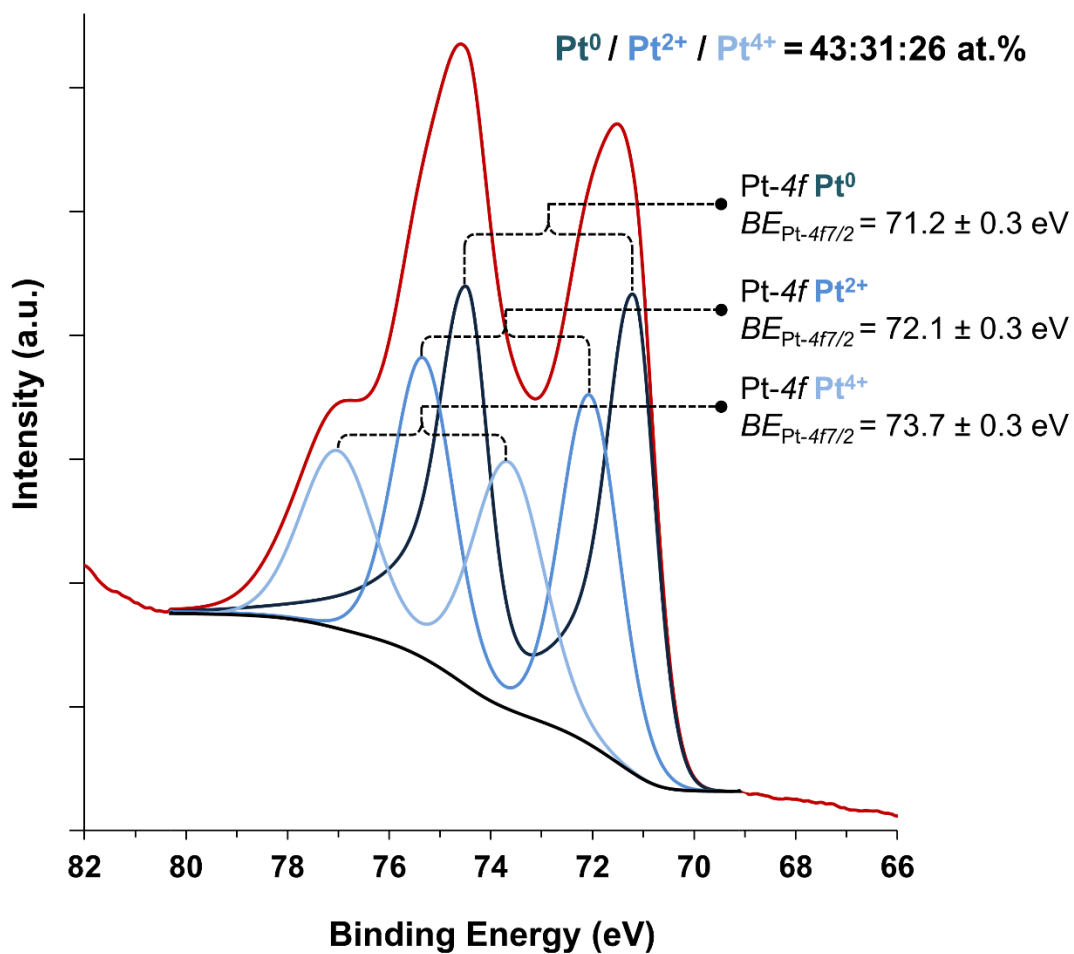


Figure S1. XPS spectrum obtained from fresh PtCNT catalyst highlighting Pt⁰, Pt²⁺ and Pt⁴⁺ contributions (Pt-4f region).

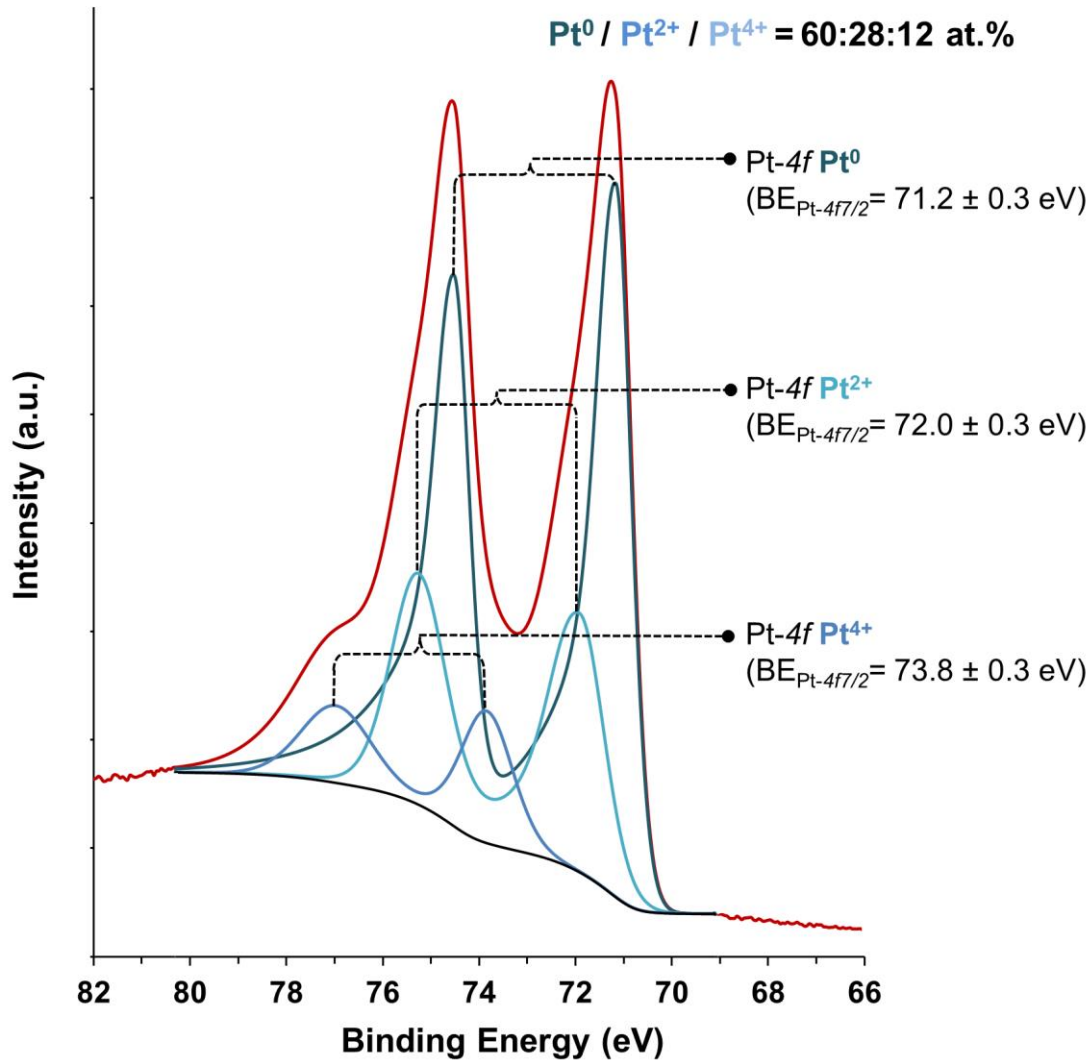


Figure S2. XPS spectrum obtained from recycled PtCNT catalyst highlighting Pt^0 , Pt^{2+} and Pt^{4+} contributions (Pt-4f region).

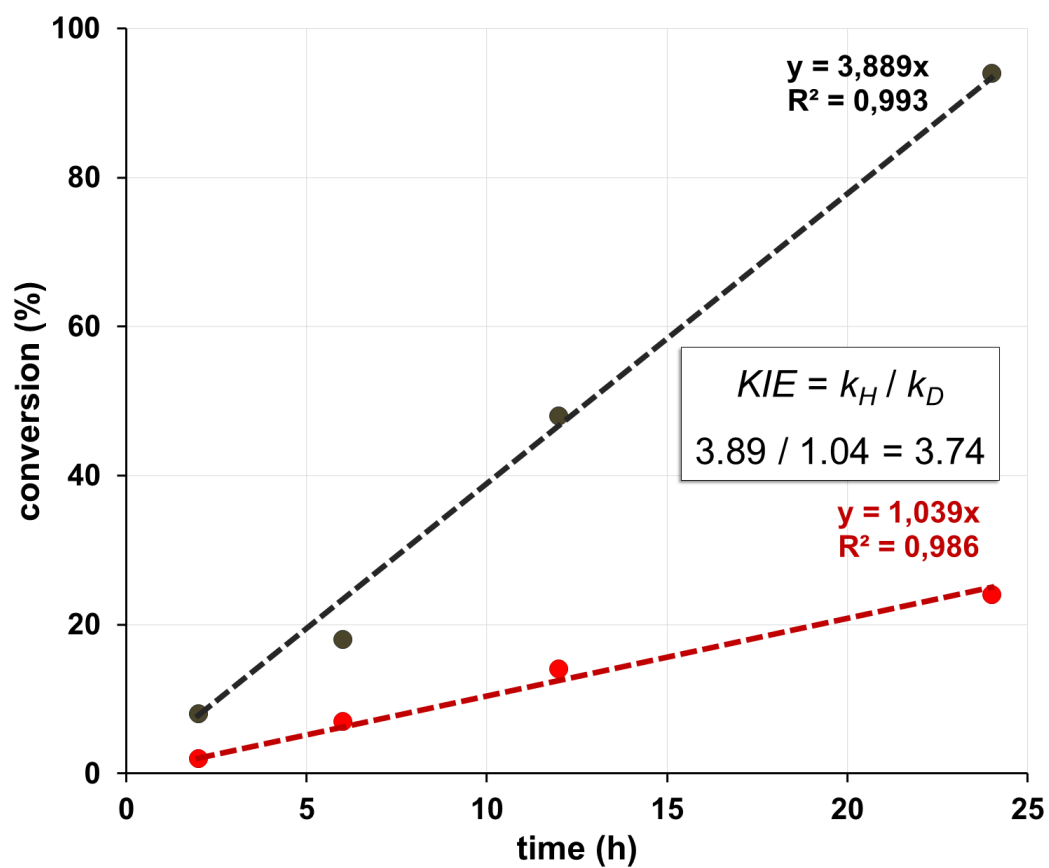
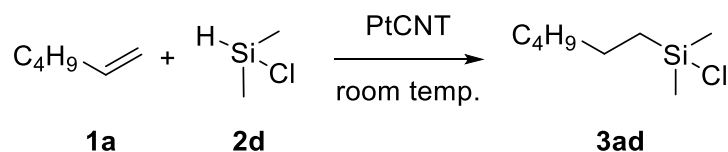


Figure S3. Determination of the kinetic isotopic effect based on the rates (k) of the reaction of hex-1-ene with HSi(Me)₂Ph (●) or DSi(Me)₂Ph (●).

4. Supplementary Table

Table S1. Recycling experiment.^a



entry	catalyst	t (h)	yield (%) ^b
1	Fresh	24	97
2	1 st reuse	24	98
3	2 nd reuse	24	97
4	3 rd reuse	24	96
5	4 th reuse	24	97
6	5 th reuse	24	96

^a **1a** (1 mmol), **2d** (1.2 mmol), neat, PtCNT (0.04 mol%), room temperature. ^b Yield of isolated product.

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