

Supporting Information

**A General and Selective Synthesis of Methylmonochlorosilanes from
Di-, Tri-, and Tetrachlorosilanes**

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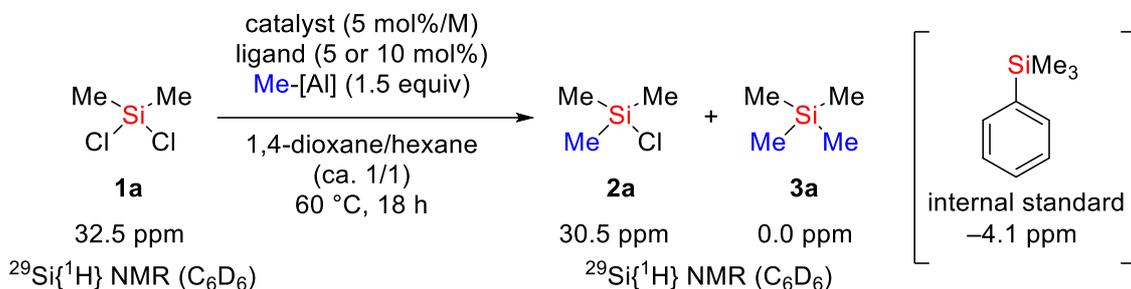
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General Information

All experiments were performed under argon atmosphere using Schlenk techniques or a glove box unless otherwise noted. 1,4-dioxane were purchased from Wako Pure Chemical Industries, Ltd. as “Super Dehydrated” and used as received. Dimethylaluminum chloride (ca. 1.0 M solution in hexane) was purchased from Kanto Chemical Co., Inc. Trimethylaluminium (ca. 1.4 M solution in hexane), triethylaluminum (1.0 M solution in hexane) and triisobutylaluminum (0.55 M solution in hexane) were purchased from Tokyo Chemical Industry Co., Ltd. Trioctylaluminium (25 wt% solution in hexane) was purchased from Sigma-Aldrich, Inc. Other reagents were purchased from commercial suppliers and used without further purification. ^1H , $^{13}\text{C}\{^1\text{H}\}$ and $^{29}\text{Si}\{^1\text{H}\}$ spectra were recorded on a Bruker AVANCE III HD 600 spectrometer. ^1H , $^{13}\text{C}\{^1\text{H}\}$ and $^{29}\text{Si}\{^1\text{H}\}$ NMR spectra (^1H , 600 MHz; ^{13}C , 150 MHz; ^{29}Si , 119 MHz) were recorded on a Bruker AVANCE 600 spectrometer. Chemical shifts are reported in δ (ppm) and referenced to the residual solvent signals for ^1H and ^{13}C , and 1,4-bis(trimethylsilyl)benzene for ^{29}Si as external standards. High-resolution ESI mass spectra were obtained on a Bruker micrOTOF II. Column chromatography was performed with silica gel (Kanto Chemical Co., Inc. Silica gel 60 N, 100-210 μm).

Optimization of reaction conditions for methylation of Me₂SiCl₂ (1a**) with methylaluminum reagents (Table 1)**

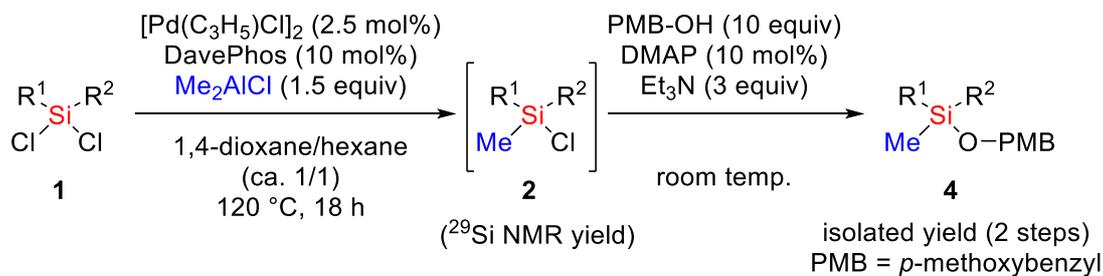


In a test tube with a PTFE screw cap, catalyst (0.025 mmol/M) and ligand (0.025 or 0.05 mmol) were equipped. 1,4-Dioxane (0.5 mL) was added into the test tube, and the solution was stirred at room temperature for ca. 5 min. Next, dichlorodimethylsilane (**1a**) (60 μL, 0.5 mmol) and dimethylaluminum chloride (750 μL, 1.0 M solution in hexane, 0.75 mmol) was successively added to the solution and the resulting mixture was stirred at 60 °C for 18 h in an oil bath. After cooling to room temperature, the conversion of **1a** and the yield of chlorotrimethylsilane (**2a**) and tetramethylsilane (**3a**) was determined by ²⁹Si{¹H} NMR spectroscopic analysis with trimethyl(phenyl)silane (86 μl, 0.5 mmol) as internal standard.

The reactions of trichloromethylsilane (**5a**) (Table 4) and tetrachlorosilane (**6**) (Scheme 3) were conducted with the similar procedure.

²⁹Si{¹H} NMR (119 MHz, CDCl₃): δ -18.4 ppm (SiCl₄), 13.1 ppm (MeSiCl₃), 32.5 ppm (Me₂SiCl₂), 30.5 ppm (Me₃SiCl), 0.0 ppm (Me₄Si), -4.1 ppm (Me₃SiPh; internal standard)

General procedure for palladium-catalyzed methylation of dichlorosilane (1**) with dimethylaluminum chloride (Table 2)**

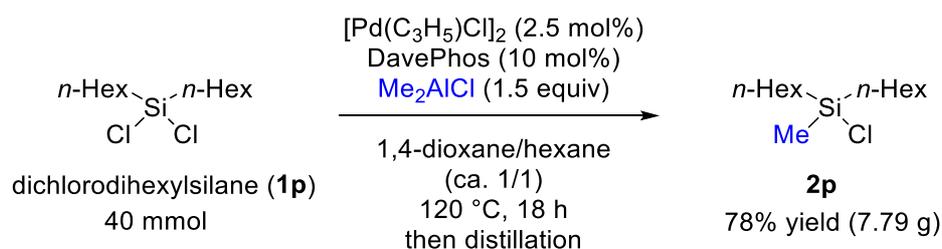


In a test tube with a PTFE screw cap, [Pd(allyl)Cl]₂ (4.6 mg, 0.0125 mmol) and DavePhos (19.7 mg, 0.05 mmol) were equipped. 1,4-Dioxane (0.5 mL) was added into

the test tube, and the solution was stirred at room temperature for ca. 5 min. Next, dichlorosilanes **1** (0.5 mmol) and dimethylaluminum chloride (750 μ L, 1.0 M solution in hexane, 0.75 mmol) was successively added to the solution and the resulting mixture was stirred at 120 $^{\circ}$ C for 18 h in an oil bath. After cooling to room temperature, the yield of **2** was determined by $^{29}\text{Si}\{^1\text{H}\}$ NMR spectroscopic analysis with trimethyl(phenyl)silane (86 μ L, 0.5 mmol) as internal standard. A series of methylmonochlorosilane **2** was identified by analogy with the trimethylchlorosilane (**2a**) as summarized in **Table S1**. After the determination of NMR yield of **2**, all the reaction mixture was treated with Et_3N (209 μ L, 1.5 mmol), DMAP (6.1 mg, 0.05 mmol) and 4-methoxybenzyl alcohol (622 μ L, 5.0 mmol), and the resulting mixture was stirred at room temperature for 1 h. Then, the mixture was diluted with hexane, filtered through a pad of Celite and the filtrate was concentrated to dryness under vacuum. The residue was purified by column chromatography on silica gel with hexane/AcOEt (40/1 to 30/1) to afford the corresponding methylalkoxysilanes **4**.

The reactions of trialkylaluminum (**Table S2**) were conducted with the similar procedure. A series of alkylmonochlorosilane **2** was identified by analogy with the trimethylchlorosilane (**2a**) as summarized in **Table S3**.

Procedure for a gram-scale synthesis of **2p** (Scheme 2).



In a Schlenk flask, $[\text{Pd}(\text{allyl})\text{Cl}]_2$ (366 mg, 1.0 mmol) and DavePhos (1574 mg, 4.0 mmol) were equipped under argon. 1,4-Dioxane (60 mL) was added and the solution was stirred at room temperature for ca. 5 min. Next, dichlorodihexylsilane (11.1 mL, 40 mmol) and dimethylaluminum chloride (60 mL, 1.0 M solution in hexane, 60 mmol) was successively added to the solution and the resulting mixture was stirred at 120 $^{\circ}$ C for 18 h in an oil bath. After the reaction, the mixture was diluted with hexane, filtered through a pad of Celite under argon and the filtrate was concentrated to dryness under vacuum. The resulting crude mixture was purified by distillation (90 $^{\circ}$ C, 0.2 Torr) to give **2p** in 78% yield (7.79 g).

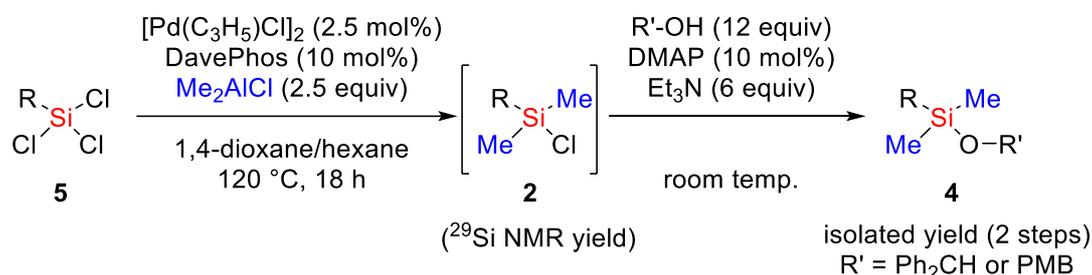
^1H NMR (600 MHz, C_6D_6): δ 1.39 (m, 4H, hexyl), 1.35-1.18 (m, 12H, hexyl), 0.89 (t, $J = 7.2$ Hz, 6H, CH_2CH_3), 0.70 (m, 4H, SiCH_2), 0.26 (s, 3H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, C_6D_6): δ 33.1, 31.8, 23.3, 22.9, 17.9, 14.3, -0.1.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, C_6D_6): δ 32.4.

The spectrum data of **2p** was in accordance with the literature.^[S1]

General procedure for palladium-catalyzed methylation of trichlorosilane (**5**) with dimethylaluminum chloride (Table 4)

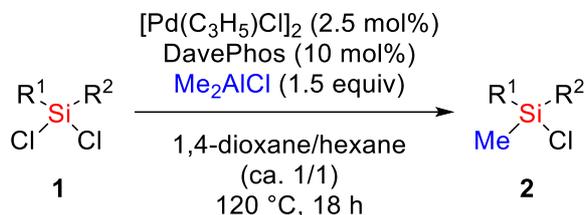


In a test tube with a PTFE screw cap, [Pd(allyl)Cl]₂ (4.6 mg, 0.0125 mmol) and DavePhos (19.7 mg, 0.05 mmol) were equipped. 1,4-Dioxane (1.0 mL) was added into the test tube, and the solution was stirred at room temperature for ca. 5 min. Next, trichlorosilanes **5** (0.5 mmol) and dimethylaluminum chloride (1.25 mL, 1.0 M solution in hexane, 1.25 mmol) was successively added to the solution and the resulting mixture was stirred at 120 °C for 18 h in an oil bath. After cooling to room temperature, yield of **2** was determined by $^{29}\text{Si}\{^1\text{H}\}$ NMR spectroscopic analysis with trimethyl(phenyl)silane (86 μL , 0.5 mmol) as internal standard. A series of dimethylmonochlorosilane **2** was identified by analogy with the trimethylchlorosilane (**2a**) as summarized in Table S4. After the determination of NMR yield of **2**, all the reaction mixture was treated with Et₃N (417 μL , 3.0 mmol), DMAP (6.1 mg, 0.05 mmol) and 4-methoxybenzyl alcohol (747 μL , 6.0 mmol), and the resulting mixture was stirred at room temperature for 1 h. Then, the mixture was diluted with hexane, filtered through a pad of Celite and the filtrate was concentrated to dryness under vacuum. The residue was purified by column chromatography on silica gel with hexane/AcOEt (40/1 to 30/1) to afford the corresponding alkyldimethylsilanes **4**.

The reactions of trialkylaluminum (Table S5) were conducted with the similar procedure. A series of alkylmonochlorosilane **2** was identified by analogy with the trimethylchlorosilane (**2a**) as summarized in Table S6.

Supporting Table

Table S1. $^{29}\text{Si}\{^1\text{H}\}$ NMR spectroscopic data (C_6D_6) for Table 2.



R^1	R^2	δ (ppm) (1) ^a	δ (ppm) (2) ^b
<i>n</i> -propyl	Me	33.0 (1b)	31.0 (2b)
<i>n</i> -hexyl	Me	33.5 (1c)	31.3 (2c)
<i>n</i> -decyl	Me	33.4 (1d)	31.3 (2d)
<i>n</i> -dodecyl	Me	33.4 (1e)	31.2 (2e)
<i>n</i> -octadecyl	Me	33.4 (1f)	31.2 (2f)
phenethyl	Me	32.8 (1g)	31.2 (2g)
3-chloropropyl	Me	32.6 (1h)	31.3 (2h)
3,3,3-trifluoropropyl	Me	31.6 (1i)	30.6 (2i)
cyclohexyl	Me	33.0 (1j)	31.3 (2j)
phenyl	Me	19.3 (1k)	20.2 (2k) ^a
phenyethenyl	Me	18.3 (1l) ^c	18.9 (2l)
ethyl	ethyl	36.5 (1m)	34.5 (2m)
<i>n</i> -butyl	<i>n</i> -butyl	34.1 (1n)	32.0 (2n)
<i>n</i> -pentyl	<i>n</i> -pentyl	34.0 (1o)	31.9 (2o)
<i>n</i> -hexyl	<i>n</i> -hexyl	34.0 (1p)	31.9 (2p)
<i>n</i> -octyl	<i>n</i> -octyl	34.0 (1q)	31.9 (2q)
2-ethylhexyl	2-ethylhexyl	33.9 (1r)	31.7 (2r)
isopropyl	isopropyl	38.5 (1s)	36.9 (2s)

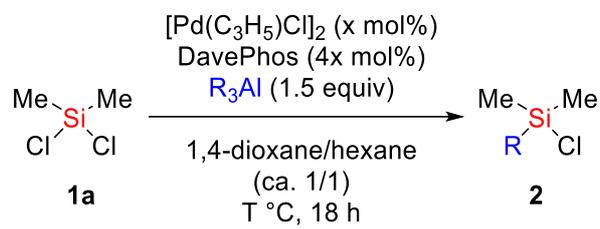
^aIdentified with a commercially available compound as reference ^bIdentified by analogy with the trimethylchlorosilane (**2a**) ^cIdentified with a compound synthesized via silyl-Heck reaction^[S2] as reference.

Table S2. Scope of palladium-catalyzed monoalkylation of **1a** with trialkylaluminum.^a

entry	x (mol%)	R ^b	T (°C)	% conv. (1a) ^c	% yield (2) ^c	R'	% yield (4) ^d
1	2.5	Et	60	>99	95 (2u)	Ph ₂ CH	92 (4u)
2	5	<i>n</i> -Bu ^d	120	>99	95 (2aa)	PMB	90 (4aa)
3	5	<i>n</i> -octyl	120	>99	88 (2v)	PMB	84 (4v)
4	5	<i>i</i> -Bu	180	>99	47 (2x)	PMB	45 (4x)

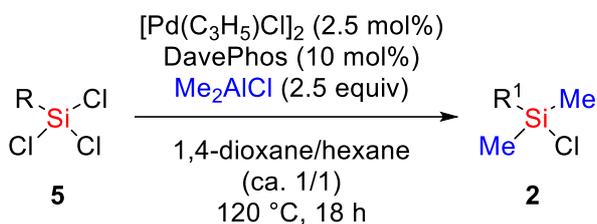
^a Reaction conditions: [Pd(C₃H₅)Cl]₂ (2.5 or 5.0 mol%), DavePhos (10 or 20 mol%), **1** (0.5 mmol), R₃Al (0.75 mmol) in dioxane (1.5 mL), 60, 120 or 180 °C, 18 h; triethylamine (1.5 mmol), 4-dimethylaminopyridine (10 mol%), *p*-methoxybenzylalcohol or diphenylmethanol (for **2u**) (5 mmol), r.t., 3 h. ^b Concentration of methylaluminum reagents: Et₃Al (1.0 M in hexane), (*n*-octyl)₃Al (25wt% in hexane), (*i*Bu)₃Al (0.55 M in hexane). ^c Determined by ²⁹Si{¹H} NMR spectroscopic analysis with trimethyl(phenyl)silane (0.5 mmol) as an internal standard. ^d Isolated yield (2 steps). ^d Prepared from AlCl₃ (0.75 mmol) and *n*-BuLi (3 equiv) in hexane (2 mL), r.t., 3 h.

Table S3. $^{29}\text{Si}\{^1\text{H}\}$ NMR spectroscopic data (C_6D_6) for Table S2.



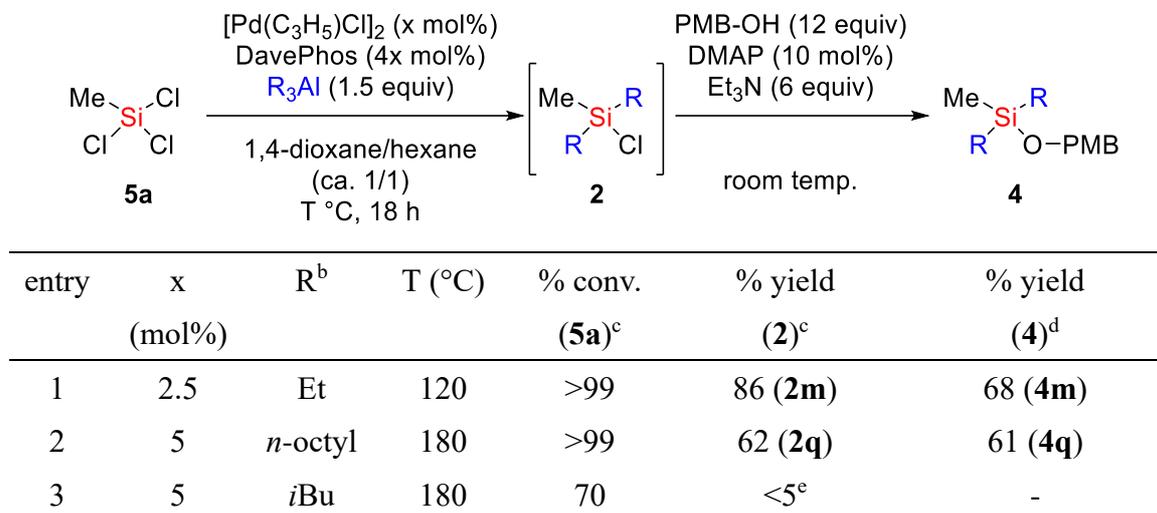
R	δ (ppm) (2) ^a
ethyl	32.7 (2u)
<i>n</i> -Bu	30.9 (2aa)
<i>n</i> -octyl	31.4 (2v)
<i>i</i> -Bu	30.5 (2x)

^aIdentified by analogy with the trimethylchlorosilane (**2a**)

Table S4. $^{29}\text{Si}\{^1\text{H}\}$ NMR spectroscopic data (C_6D_6) for Table 4.

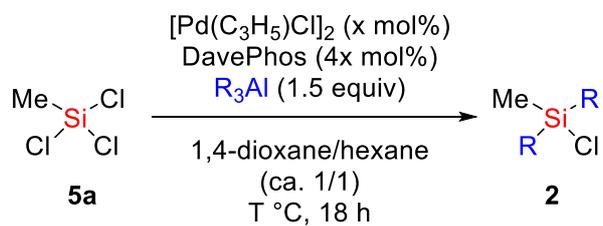
R	δ (ppm) (5) ^a	δ (ppm) (2) ^b
ethyl	15.0 (5u)	32.7 (2u)
<i>n</i> -propyl	13.5 (5b)	31.1 (2b)
<i>n</i> -octyl	13.8 (5v)	31.1 (2v)
<i>n</i> -decyl	13.8 (5d)	31.3 (2d)
<i>n</i> -dodecyl	13.8 (5e)	31.3 (2e)
<i>n</i> -hexadecyl	13.8 (5w)	31.3 (2w)
<i>n</i> -octadecyl	13.8 (5f)	31.3 (2f)
isobutyl	12.9 (5x)	30.5 (2x)
phenethyl	12.8 (5g)	31.3 (2g)
3-chloropropyl	12.9 (5h)	31.4 (2h)
3,3,3-trifluoropropyl	11.6 (5i)	31.2 (2i)
1 <i>H</i> ,1 <i>H</i> ,2 <i>H</i> ,2 <i>H</i> -tridecafluoro- <i>n</i> -octyl	11.9 (5y)	31.5 (2y)
cyclopentyl	14.1 (5z)	31.9 (2z)
cyclohexyl	13.5 (5j)	31.4 (2j)
phenyl	-0.3 (5k)	20.3 (2k) ^a

^aIdentified with a commercially available compound as reference ^bIdentified by analogy with the trimethylchlorosilane (**2a**)

Table S5. Scope of palladium-catalyzed double alkylation of **5a** with trialkylaluminum.^a

^a Reaction conditions: [Pd(C₃H₅)Cl]₂ (2.5 or 5.0 mol%), DavePhos (10 or 20 mol%), **1** (0.5 mmol), R₃Al (0.75 mmol) in dioxane (1.5 mL), 60 or 120 °C, 18 h; then triethylamine (3 mmol), 4-dimethylaminopyridine (10 mol%), *p*-methoxybenzylalcohol (6 mmol), r.t., 3 h. ^b Concentration of methylaluminum reagents: Et₃Al (1.0 M in hexane), (*n*-octyl)₃Al (25wt% in hexane), (*i*Bu)₃Al (0.55 M in hexane). ^c Determined by ²⁹Si{¹H} NMR spectroscopic analysis with trimethyl(phenyl)silane (0.5 mmol) as an internal standard. ^d Isolated yield (2 steps). ^e *i*BuMeSiCl₂ was observed in 39% NMR yield.

Table S6. $^{29}\text{Si}\{^1\text{H}\}$ NMR spectroscopic data (C_6D_6) for Table S5.

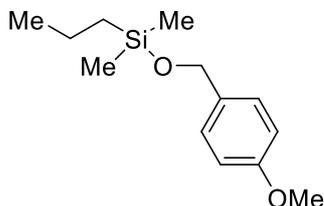


R	δ (ppm) (2)
ethyl	34.6 (2m) ^a
<i>n</i> -octyl	32.0 (2q) ^a
<i>i</i> Bu	32.4 (<i>i</i> BuMeSiCl ₂) ^b

^aIdentified by analogy with the trimethylchlorosilane (**2a**)
^bIdentified by analogy with the dimethyldichlorosilane (**1a**) and ^1H NMR analysis

Spectral Data

(4-methoxybenzyloxy)dimethyl(*n*-propyl)silane (4b)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 30/1), obtained as colorless oil (87% yield, 103.6 mg).

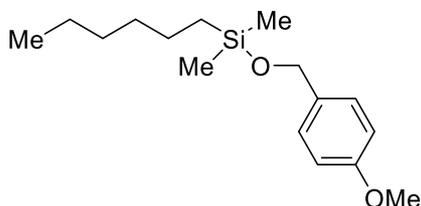
^1H NMR (600 MHz, CDCl_3): δ 7.24 (d, $J = 8.6$ Hz, 2H, Ar), 6.87 (d, $J = 8.6$ Hz, 2H, Ar), 4.63 (s, 2H, OCH_2Ar), 3.80 (s, 3H, OCH_3), 1.39 (m, 2H, $\text{CH}_2\text{CH}_2\text{CH}_3$), 0.96 (t, $J = 7.3$ Hz, 3H, CH_2CH_3), 0.63 (m, 2H, SiCH_2), 0.12 (s, 6H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.8, 133.2, 128.0, 113.7, 64.4, 55.3, 19.0, 18.1, 16.7, -1.9.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 18.6.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{22}\text{O}_2\text{SiNa}$: 261.1281; Found: 261.1291.

(4-methoxybenzyloxy)dimethyl(*n*-hexyl)silane (4c)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 30/1), obtained as colorless oil (91% yield, 127.8 mg).

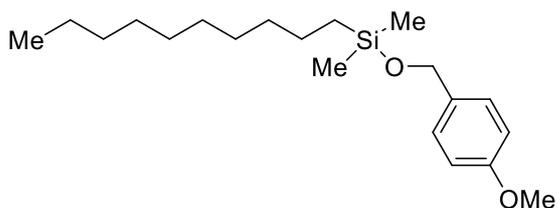
^1H NMR (600 MHz, CDCl_3): δ 7.24 (d, $J = 8.6$ Hz, 2H, Ar), 6.87 (d, $J = 8.6$ Hz, 2H, Ar), 4.63 (s, 2H, OCH_2Ar), 3.80 (s, 3H, OCH_3), 1.40-1.20 (m, 8H, hexyl), 0.88 (t, $J = 7.2$ Hz, 3H, CH_2CH_3), 0.62 (m, 2H, SiCH_2), 0.11 (s, 6H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.8, 133.2, 128.0, 113.7, 64.4, 55.3, 33.1, 31.6, 23.1, 22.6, 16.4, 14.1, -2.0.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 18.8.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{16}\text{H}_{28}\text{O}_2\text{SiNa}$: 303.1751; Found: 303.1756.

(4-methoxybenzyloxy)dimethyl(*n*-decyl)silane (4d)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 30/1), obtained as colorless oil (89% yield, 150.0 mg).

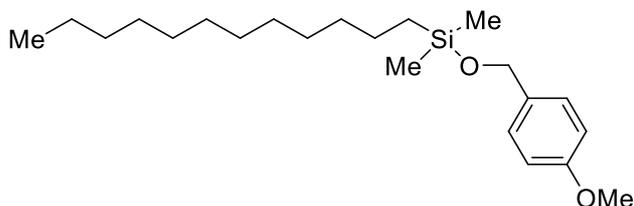
^1H NMR (600 MHz, CDCl_3): δ 7.24 (d, $J = 8.7$ Hz, 2H, Ar), 6.87 (d, $J = 8.7$ Hz, 2H, Ar), 4.63 (s, 2H, OCH_2Ar), 3.80 (s, 3H, OCH_3), 1.40-1.20 (m, 16H, decyl), 0.88 (t, $J = 7.2$ Hz, 3H, CH_2CH_3), 0.61 (m, 2H, SiCH_2), 0.11 (s, 6H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.8, 133.3, 128.1, 113.7, 64.4, 55.3, 33.5, 32.0, 29.7, 29.6, 29.4 (2C were overlapped.), 23.2, 22.7, 16.4, 14.2, -2.0.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 18.9.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{20}\text{H}_{36}\text{O}_2\text{SiNa}$: 359.2377; Found: 359.2374.

(4-methoxybenzyloxy)dimethyl(*n*-dodecyl)silane (4e)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 40/1), obtained as colorless oil (89% yield, 162.8 mg).

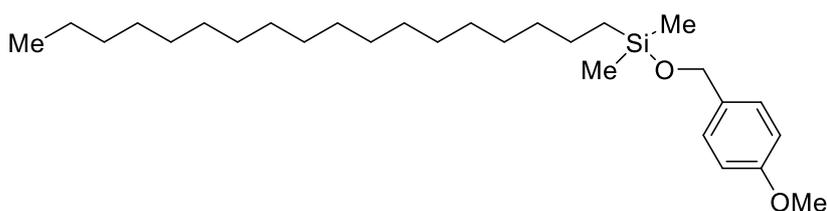
^1H NMR (600 MHz, CDCl_3): δ 7.24 (d, $J = 8.6$ Hz, 2H, Ar), 6.86 (d, $J = 8.6$ Hz, 2H, Ar), 4.63 (s, 2H, OCH_2Ar), 3.80 (s, 3H, OCH_3), 1.40-1.20 (m, 20H, dodecyl), 0.88 (t, $J = 7.2$ Hz, 3H, CH_2CH_3), 0.62 (m, 2H, SiCH_2), 0.11 (s, 6H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.8, 133.3, 128.1, 113.7, 64.4, 55.3, 33.5, 32.0, 29.74, 29.72, 29.68, 29.62, 29.4 (2C were overlapped.), 23.2, 22.7, 16.4, 14.2, -2.0.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 18.9.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{22}\text{H}_{40}\text{O}_2\text{SiNa}$: 387.2690; Found: 387.2685.

(4-methoxybenzyloxy)dimethyl(*n*-octadecyl)silane (4f)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 30/1), obtained as colorless oil (91% yield, 205.3 mg).

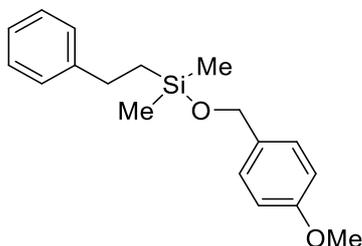
^1H NMR (600 MHz, CDCl_3): δ 7.24 (d, J = 8.6 Hz, 2H, Ar), 6.87 (d, J = 8.6 Hz, 2H, Ar), 4.63 (s, 2H, OCH_2Ar), 3.80 (s, 3H, OCH_3), 1.40-1.20 (m, 32H, octadecyl), 0.88 (t, J = 7.2 Hz, 3H, CH_2CH_3), 0.61 (m, 2H, SiCH_2), 0.11 (s, 6H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.8, 133.3, 128.1, 113.7, 64.4, 55.3, 33.5, 32.0, 29.73 (8C were overlapped.), 29.69, 29.62, 29.4 (2C were overlapped.), 23.2, 22.7, 16.4, 14.2, -2.0.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 18.9.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{28}\text{H}_{52}\text{O}_2\text{SiNa}$: 471.3629; Found: 471.3631.

(4-methoxybenzyloxy)dimethyl(phenethyl)silane (4g)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 30/1), obtained as colorless oil (85% yield, 127.4 mg).

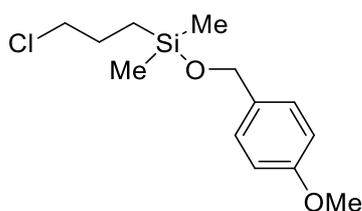
^1H NMR (600 MHz, CDCl_3): δ 7.30-7.22 (m, 4H, Ar), 7.21-7.13 (m, 3H, Ar), 6.88 (m, 2H, Ar), 4.65 (s, 2H, OCH_2Ar), 3.81 (s, 3H, OCH_3), 2.67 (m, 2H, $\text{CH}_2\text{CH}_2\text{Ph}$), 0.99 (m, 2H, SiCH_2), 0.15 (s, 6H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.9, 144.9, 133.1, 128.3, 128.1, 127.8, 125.6, 113.8, 64.5, 55.3, 29.3, 18.5, -2.0.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 18.2.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{18}\text{H}_{24}\text{O}_2\text{SiNa}$: 323.1438; Found: 323.1437.

(4-methoxybenzyloxy)dimethyl(3-chloropropyl)silane (4h)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 30/1), obtained as colorless oil (86% yield, 116.9 mg).

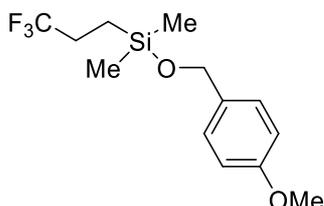
^1H NMR (600 MHz, CDCl_3): δ 7.24 (d, J = 8.7 Hz, 2H, Ar), 6.87 (d, J = 8.7 Hz, 2H, Ar), 4.63 (s, 2H, OCH_2Ar), 3.80 (s, 3H, OCH_3), 3.49 (t, J = 6.9 Hz, 2H, ClCH_2), 1.80 (m, 2H, $\text{CH}_2\text{CH}_2\text{CH}_2$), 0.72 (m, 2H, SiCH_2), 0.14 (s, 6H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.9, 132.9, 128.1, 113.8, 64.6, 55.3, 47.9, 26.9, 14.1, -2.0.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 18.2.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{21}\text{ClO}_2\text{SiNa}$: 295.0892; Found: 295.0900.

(4-methoxybenzyloxy)dimethyl(3,3,3-trifluoropropyl)silane (4i)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 40/1), obtained as colorless oil (82% yield, 120.0 mg).

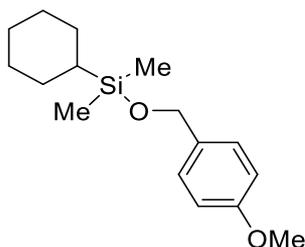
^1H NMR (600 MHz, CDCl_3): δ 7.23 (d, J = 8.7 Hz, 2H, Ar), 6.88 (d, J = 8.7 Hz, 2H, Ar), 4.63 (s, 2H, OCH_2Ar), 3.81 (s, 3H, OCH_3), 2.04 (m, 2H, CF_3CH_2), 0.81 (m, 2H, SiCH_2), 0.16 (s, 6H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 159.0, 132.6, 128.2, 127.7 (q, $^1J_{\text{C-F}}$ = 276.4 Hz), 113.8, 64.7, 55.3, 28.1 (q, $^2J_{\text{C-F}}$ = 30.0 Hz), 8.3 (q, $^3J_{\text{C-F}}$ = 1.9 Hz), -2.2.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 17.5.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{19}\text{F}_3\text{O}_2\text{SiNa}$: 315.0999; Found: 315.0987.

(4-methoxybenzyloxy)dimethyl(cyclohexyl)silane (4j)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 20/1), obtained as colorless oil (91% yield, 126.1mg).

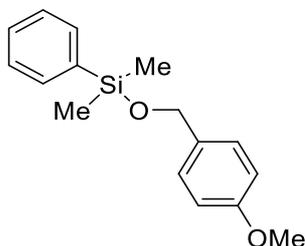
^1H NMR (600 MHz, CDCl_3): δ 7.24 (d, $J = 8.7$ Hz, 2H, Ar), 6.87 (d, $J = 8.7$ Hz, 2H, Ar), 4.63 (s, 2H, OCH_2Ar), 3.80 (s, 3H, OCH_3), 1.80-1.60 (m, 5H, Cy), 1.30-1.00 (m, 5H, Cy), 0.74 (m, 1H, SiCH), 0.07 (s, 6H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.7, 133.4, 127.9, 113.7, 64.5, 55.3, 27.9, 27.0, 26.8, 26.7, -4.0.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 17.9.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{16}\text{H}_{26}\text{O}_2\text{SiNa}$: 301.1594; Found: 301.1604.

(4-methoxybenzyloxy)dimethyl(phenyl)silane (4k)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 30/1), obtained as colorless oil (85% yield, 115.2 mg).

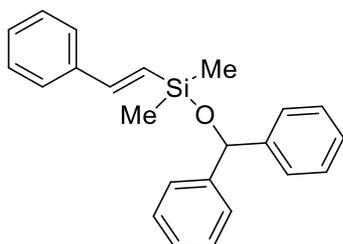
^1H NMR (600 MHz, CDCl_3): δ 7.60 (m, 2H, Ar), 7.40 (m, 3H, Ar), 7.21 (d, $J = 8.7$ Hz, 2H, Ar), 6.86 (d, $J = 8.7$ Hz, 2H, Ar), 4.63 (s, 2H, OCH_2Ar), 3.80 (s, 3H, OCH_3), 0.40 (s, 6H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.9, 137.7, 133.6, 132.9, 129.7, 128.2, 127.9, 113.7, 64.8, 55.3, -1.6.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 8.5.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{16}\text{H}_{20}\text{O}_2\text{SiNa}$: 295.1125; Found: 295.1126.

(benzhydryloxy)dimethyl(phenylethenyl)silane (4l)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 30/1), obtained as colorless oil (63% yield, 109.0 mg).

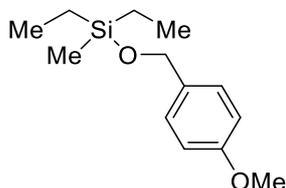
^1H NMR (600 MHz, CDCl_3): δ 7.42-7.18 (m, 15H, Ph), 6.90 (dd, $J = 19.2, 2.0$ Hz, 1H, $\text{PhCH}=\text{CH}$), 6.32 (dd, $J = 19.2, 2.6$ Hz, 1H, $\text{PhCH}=\text{CH}$), 5.83 (s, 1H, OCH), 0.22 (s, 6H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 145.6, 144.7, 138.0, 128.5, 128.3, 128.2, 127.1, 126.64, 126.61, 126.55, 76.8, -1.1.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 7.7.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{23}\text{H}_{24}\text{OSiNa}$: 367.1489; Found: 367.1496.

(4-methoxybenzyloxy)methyl(diethyl)silane (4m)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 20/1), obtained as colorless oil (95% yield, 113.3 mg).

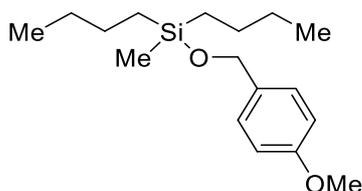
^1H NMR (600 MHz, CDCl_3): δ 7.25 (d, $J = 8.7$ Hz, 2H, Ar), 6.87 (d, $J = 8.7$ Hz, 2H, Ar), 4.64 (s, 2H, OCH_2Ar), 3.80 (s, 3H, OCH_3), 0.96 (t, $J = 7.9$ Hz, 6H, CH_2CH_3), 0.62 (m, 4H, SiCH_2), 0.09 (s, 3H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.8, 133.4, 127.9, 113.7, 64.5, 55.3, 6.8, 6.3, -4.8.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 20.5.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{22}\text{O}_2\text{SiNa}$: 261.1281; Found: 261.1286.

(4-methoxybenzyloxy)methyl(di-*n*-butyl)silane (4n)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 30/1), obtained as colorless oil (88% yield, 129.2 mg).

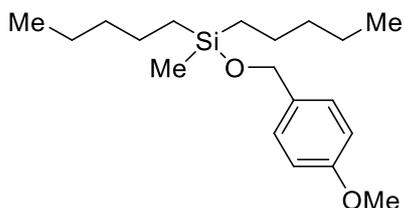
^1H NMR (600 MHz, CDCl_3): δ 7.24 (d, $J = 8.8$ Hz, 2H, Ar), 6.87 (d, $J = 8.7$ Hz, 2H, Ar), 4.63 (s, 2H, OCH_2Ar), 3.80 (s, 3H, OCH_3), 1.35-1.26 (m, 8H, butyl), 0.88 (t, $J = 7.1$ Hz, 6H, CH_2CH_3), 0.62 (m, 4H, SiCH_2), 0.09 (s, 3H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.8, 133.4, 127.9, 113.7, 64.4, 55.3, 26.5, 25.4, 14.8, 13.8, -3.8.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 18.7.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{17}\text{H}_{30}\text{O}_2\text{SiNa}$: 317.1907; Found: 317.1909.

(4-methoxybenzyloxy)methyl(di-*n*-pentyl)silane (4o)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 20/1), obtained as colorless oil (96% yield, 155.5 mg).

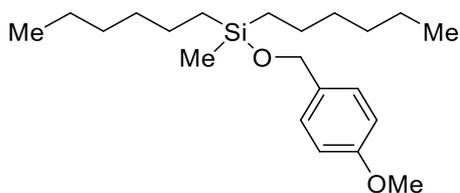
^1H NMR (600 MHz, CDCl_3): δ 7.24 (d, $J = 8.7$ Hz, 2H, Ar), 6.86 (d, $J = 8.7$ Hz, 2H, Ar), 4.63 (s, 2H, OCH_2Ar), 3.80 (s, 3H, OCH_3), 1.40-1.20 (m, 12H, pentyl), 0.87 (t, $J = 7.0$ Hz, 6H, CH_2CH_3), 0.61 (m, 4H, SiCH_2), 0.09 (s, 3H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.8, 133.4, 127.9, 113.7, 64.4, 55.3, 35.8, 22.8, 22.3, 15.1, 14.0, -3.8.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 18.6.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{19}\text{H}_{34}\text{O}_2\text{SiNa}$: 345.2220; Found: 345.2213.

(4-methoxybenzyloxy)methyl(di-*n*-hexyl)silane (4p)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 20/1), obtained as colorless oil (94% yield, 165.1 mg).

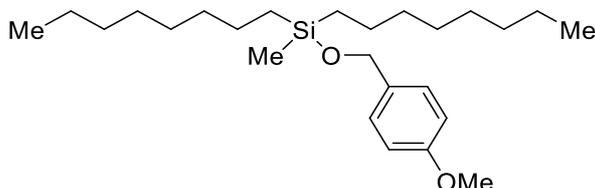
^1H NMR (600 MHz, CDCl_3): δ 7.24 (d, $J = 8.2$ Hz, 2H, Ar), 6.86 (d, $J = 8.2$ Hz, 2H, Ar), 4.63 (s, 2H, OCH_2Ar), 3.80 (s, 3H, OCH_3), 1.40-1.15 (m, 16H, hexyl), 0.88 (t, $J = 7.2$ Hz, 6H, CH_2CH_3), 0.60 (m, 4H, SiCH_2), 0.09 (s, 3H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.8, 133.4, 128.0, 113.7, 64.4, 55.3, 33.2, 31.6, 23.2, 22.6, 15.1, 14.2, -3.8.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 18.6.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{21}\text{H}_{38}\text{O}_2\text{SiNa}$: 373.2533; Found: 373.2538.

(4-methoxybenzyloxy)methyl(di-*n*-octyl)silane (4q)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 30/1), obtained as colorless oil (80% yield, 161.9 mg).

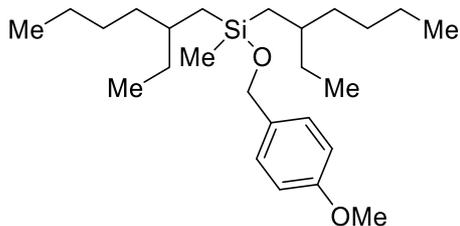
^1H NMR (600 MHz, CDCl_3): δ 7.24 (d, $J = 8.7$ Hz, 2H, Ar), 6.86 (d, $J = 8.7$ Hz, 2H, Ar), 4.63 (s, 2H, OCH_2Ar), 3.80 (s, 3H, OCH_3), 1.40-1.20 (m, 24H, octyl), 0.88 (t, $J = 7.1$ Hz, 6H, CH_2CH_3), 0.62 (m, 4H, SiCH_2), 0.09 (s, 3H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.8, 133.4, 128.0, 113.7, 64.4, 55.3, 33.6, 32.0, 29.31, 29.28, 23.2, 22.7, 15.1, 14.1, -3.8.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 18.7.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{25}\text{H}_{46}\text{O}_2\text{SiNa}$: 429.3159; Found: 429.3145.

(4-methoxybenzyloxy)methyl(di-2-ethylhexyl)silane (4r)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 30/1), obtained as colorless oil (77% yield, 156.2 mg).

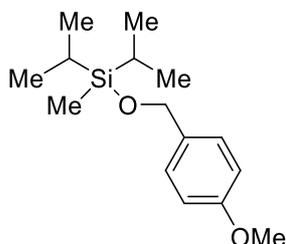
^1H NMR (600 MHz, CDCl_3): δ 7.24 (d, $J = 8.5$ Hz, 2H, Ar), 6.86 (d, $J = 8.6$ Hz, 2H, Ar), 4.63 (s, 2H, OCH_2Ar), 3.80 (s, 3H, OCH_3), 1.49 (m, 2H, CH_2CH), 1.40-1.15 (m, 16H, 2-ethylhexyl), 0.88 (t, $J = 7.1$ Hz, 6H, CH_2CH_3), 0.83 (t, $J = 7.4$ Hz, 6H, CH_2CH_3), 0.62 (m, 4H, SiCH_2), 0.15 (s, 3H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.7, 133.5, 127.8, 113.6, 64.2, 55.3, 35.7*, 34.8*, 29.0, 28.8, 23.1, 20.7*, 14.2, 10.8*, -2.0. (* Splitting due to the corresponding diastereomers)

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 18.5.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{25}\text{H}_{46}\text{O}_2\text{SiNa}$: 429.3159; Found: 429.3163.

(4-methoxybenzyloxy)methyl(diisopropyl)silane (4s)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 40/1), obtained as colorless oil (31% yield, 41.3 mg).

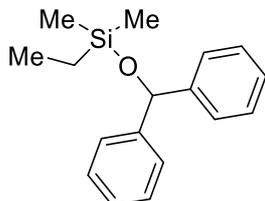
^1H NMR (600 MHz, CDCl_3): δ 7.25 (d, $J = 8.7$ Hz, 2H, Ar), 6.87 (d, $J = 8.7$ Hz, 2H, Ar), 4.69 (s, 2H, OCH_2Ar), 3.80 (s, 3H, OCH_3), 1.06-0.96 (m, 14H, *i*-Pr), 0.05 (s, 3H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.7, 133.6, 127.6, 113.6, 64.8, 55.3, 17.5, 17.4, 13.1, -8.5.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 19.3.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{15}\text{H}_{26}\text{O}_2\text{SiNa}$: 289.1594; Found: 289.1587.

(benzhydryloxy)dimethyl(ethyl)silane (4u)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 40/1), obtained as colorless oil (92% yield, 124.0 mg).

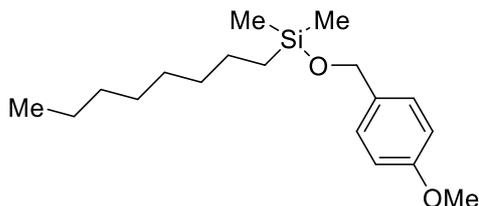
^1H NMR (600 MHz, CDCl_3): δ 7.34 (m, 4H, Ar), 7.29 (m, 4H, Ar), 7.21 (m, 2H, Ar), 5.75 (s, 1H, CHPh_2), 0.89 (t, $J = 8.0$ Hz, 3H, CH_2CH_3), 0.54 (q, $J = 8.0$ Hz, 2H, SiCH_2), 0.04 (s, 6H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 145.0, 128.2, 127.0, 126.5, 76.4, 8.5, 6.7, -2.1.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 19.5.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{17}\text{H}_{22}\text{OSiNa}$: 293.1332; Found: 293.1342.

(4-methoxybenzyloxy)dimethyl(octyl)silane (4v)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 30/1), obtained as colorless oil (91% yield, 140.0 mg).

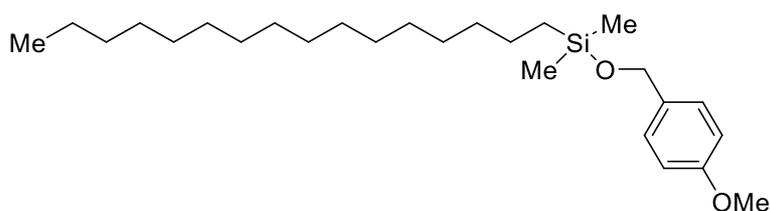
^1H NMR (600 MHz, CDCl_3): δ 7.24 (d, $J = 8.6$ Hz, 2H, Ar), 6.86 (d, $J = 8.6$ Hz, 2H, Ar), 4.62 (s, 2H, OCH_2Ar), 3.80 (s, 3H, OCH_3), 1.40-1.20 (m, 12H, octyl), 0.88 (t, $J = 7.1$ Hz, 3H, CH_2CH_3), 0.61 (m, 2H, SiCH_2), 0.11 (s, 6H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.8, 133.2, 128.1, 113.7, 64.4, 55.3, 33.5, 32.0, 29.34, 29.27, 23.2, 22.7, 16.4, 14.1, -2.0.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 18.9.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{18}\text{H}_{32}\text{O}_2\text{SiNa}$: 331.2064; Found: 331.2070.

(4-methoxybenzyloxy)dimethyl(*n*-hexadecyl)silane (4w)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 40/1), obtained as colorless oil (93% yield, 195.4 mg).

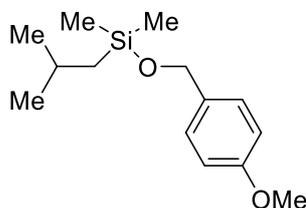
^1H NMR (600 MHz, CDCl_3): δ 7.24 (d, $J = 8.6$ Hz, 2H, Ar), 6.86 (d, $J = 8.6$ Hz, 2H, Ar), 4.62 (s, 2H, OCH_2Ar), 3.80 (s, 3H, OCH_3), 1.40-1.20 (m, 28H, hexadecyl), 0.88 (t, $J = 7.0$ Hz, 3H, CH_2CH_3), 0.61 (m, 2H, SiCH_2), 0.11 (s, 6H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.8, 133.2, 128.1, 113.7, 64.4, 55.3, 33.5, 32.0, 29.73 (6C were overlapped.), 29.69, 29.6, 29.4 (2C were overlapped.), 23.2, 22.7, 16.4, 14.2, -2.0.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 18.9.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{26}\text{H}_{48}\text{O}_2\text{SiNa}$: 443.3316; Found: 443.3324.

(4-methoxybenzyloxy)dimethyl(isobutyl)silane (4x)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 40/1), obtained as colorless oil (94% yield, 118.6 mg).

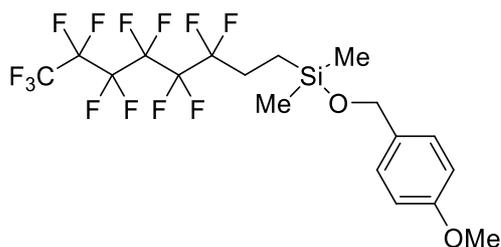
^1H NMR (600 MHz, CDCl_3): δ 7.24 (d, $J = 8.7$ Hz, 2H, Ar), 6.86 (d, $J = 8.7$ Hz, 2H, Ar), 4.62 (s, 2H, OCH_2Ar), 3.80 (s, 3H, OCH_3), 1.83 (m, 1H, CHCH_3), 0.95 (d, $J = 6.6$ Hz, 6H, CHCH_3), 0.64 (d, $J = 7.0$ Hz, 2H, SiCH_2), 0.14 (s, 6H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.8, 133.3, 128.0, 113.7, 64.3, 55.3, 27.0, 26.2, 24.3, -1.1.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 18.2.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{14}\text{H}_{24}\text{O}_2\text{SiNa}$: 275.1438; Found: 275.1449.

(4-methoxybenzyloxy)dimethyl(1H,1H,2H,2H-tridecafluoro-*n*-octyl)silane (4y)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 40/1), obtained as colorless oil (77% yield, 208.8 mg).

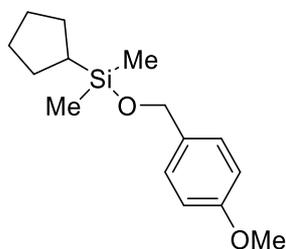
^1H NMR (600 MHz, CDCl_3): δ 7.23 (d, $J = 8.6$ Hz, 2H, Ar), 6.87 (d, $J = 8.6$ Hz, 2H, Ar), 4.64 (s, 2H, OCH_2Ar), 3.80 (s, 3H, OCH_3), 2.02 (m, 2H, CH_2CF_2), 0.81 (m, 2H, SiCH_2), 0.17 (s, 6H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 159.1, 132.5, 128.2, 113.8, 64.7, 55.3, 25.1 (t, $^2J_{\text{C-F}} = 23.9$ Hz), 6.0, -2.2. (5C were not assignable due to the complicated ^{13}C - ^{19}F coupling.)

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 17.9.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{18}\text{H}_{19}\text{F}_{13}\text{O}_2\text{SiNa}$: 565.0839; Found: 565.0831.

(4-methoxybenzyloxy)dimethyl(cyclopentyl)silane (4z)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 30/1), obtained as colorless oil (88% yield, 116.8 mg).

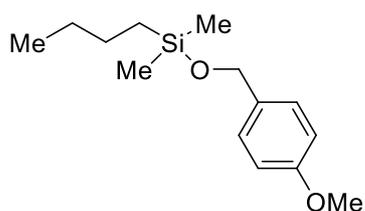
^1H NMR (600 MHz, CDCl_3): δ 7.27 (d, $J = 8.8$ Hz, 2H, Ar), 6.89 (d, $J = 8.8$ Hz, 2H, Ar), 4.68 (s, 2H, OCH_2Ar), 3.82 (s, 3H, OCH_3), 1.78 (m, 2H, Cy), 1.67-1.47 (m, 4H, Cy), 1.38 (m, 2H, Cy), 1.05 (m, 1H, SiCH), 0.12 (s, 6H, SiCH_3).

$^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 158.8, 133.4, 127.9, 113.7, 64.5, 55.3, 27.5, 27.1, 26.1, -3.5.

$^{29}\text{Si}\{^1\text{H}\}$ NMR (119 MHz, CDCl_3): δ 18.8.

HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{15}\text{H}_{24}\text{O}_2\text{SiNa}$: 287.1438; Found: 287.1445.

(4-methoxybenzyloxy)dimethyl(*n*-butyl)silane (4aa)



Purified by silica gel chromatography (eluent; hexane/AcOEt = 30/1), obtained as colorless oil (90% yield, 113.3 mg).

¹H NMR (600 MHz, CDCl₃): δ 7.24 (d, *J* = 7.8 Hz, 2H, Ar), 6.87 (d, *J* = 7.8 Hz, 2H, Ar), 4.62 (s, 2H, OCH₂Ar), 3.80 (s, 3H, OCH₃), 1.31 (m, 2H, CH₂CH₂CH₂CH₃), 0.88 (t, *J* = 6.6 Hz, 3H, CH₂CH₃), 0.62 (m, 2H, SiCH₂), 0.11 (s, 6H, SiCH₃).

¹³C{¹H} NMR (150 MHz, CDCl₃): δ 158.8, 133.3, 128.1, 113.7, 64.4, 55.3, 26.4, 25.4, 16.1, 13.8, -1.9.

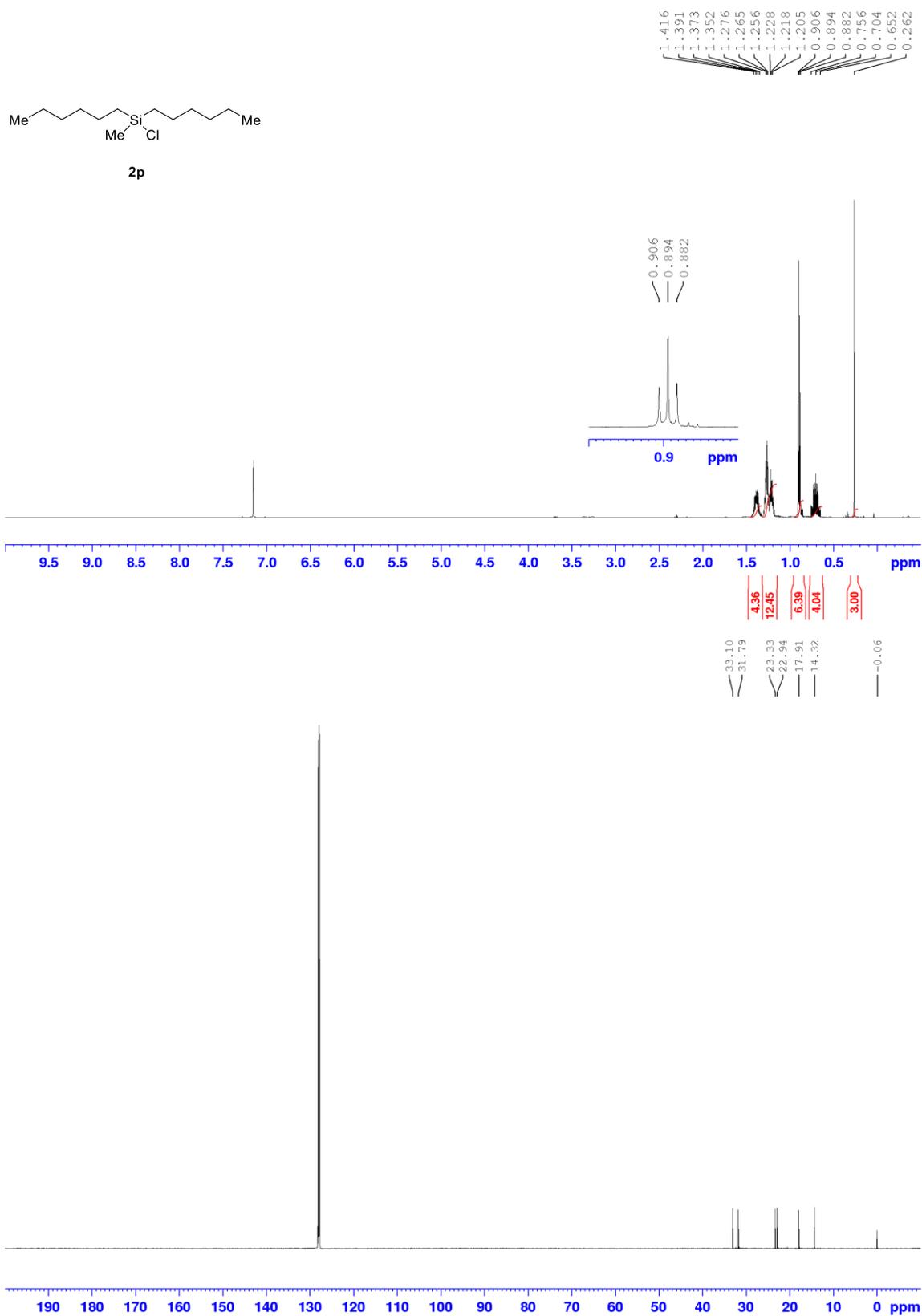
²⁹Si{¹H} NMR (119 MHz, CDCl₃): δ 18.9.

HRMS (ESI) *m/z*: [M + Na]⁺ Calcd for C₁₄H₂₄O₂SiNa: 275.1438; Found: 275.1449.

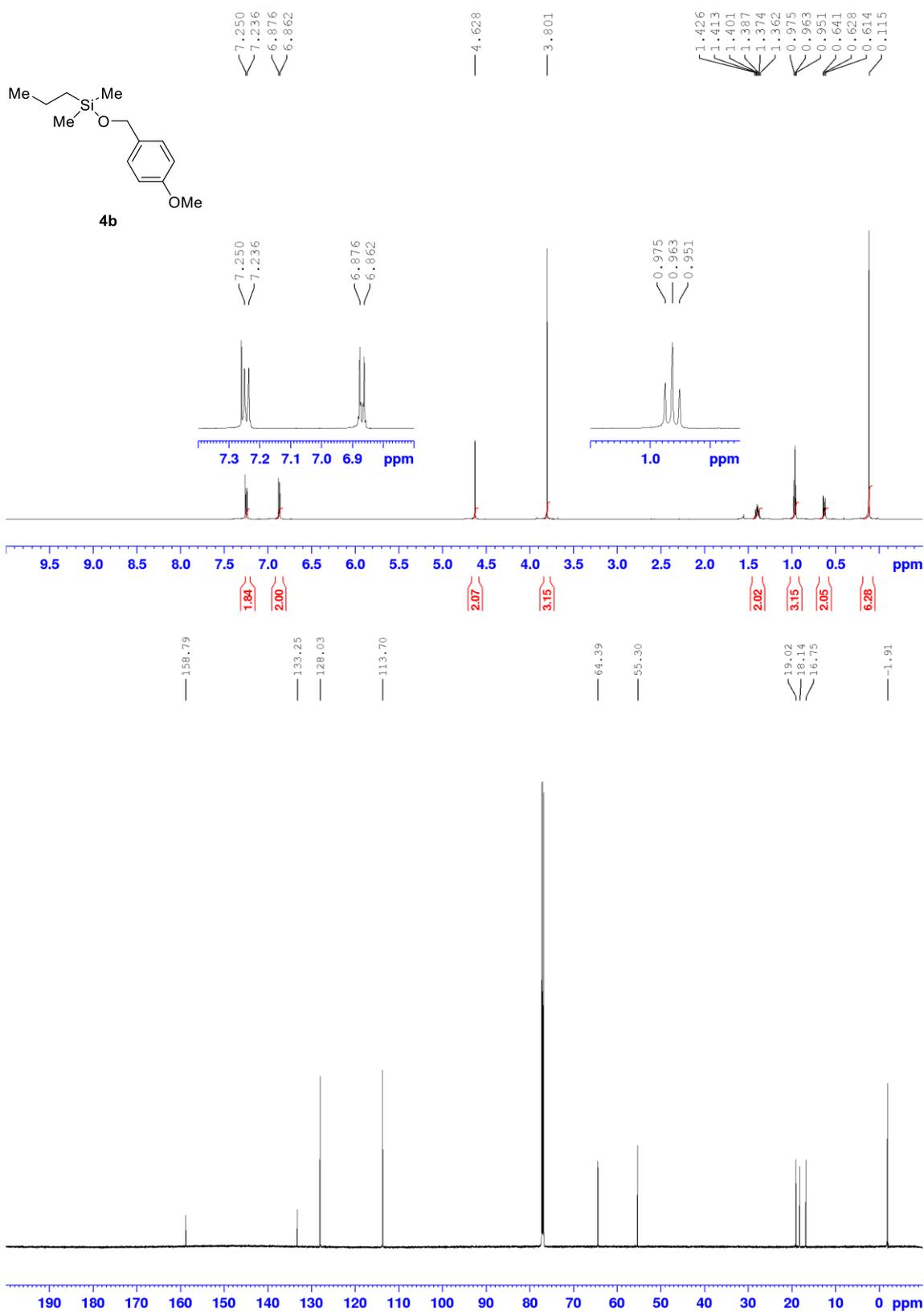
References

- [S1] Ohmura, T.; Torigoe, T.; Suginome, M. Catalytic Functionalization of Methyl Group on Silicon: Iridium-Catalyzed C(sp³)-H Borylation of Methylchlorosilanes. *J. Am. Chem. Soc.* **2012**, *134*, 17416-17419.
- [S2] Matsumoto, K.; Huang, J.; Naganawa, Y.; Guo, H.; Beppu, T.; Sato, K.; Shimada, S.; Nakajima, Y. Direct Silyl-Heck Reaction of Chlorosilanes. *Org. Lett.* **2018**, *20*, 2481-2484.

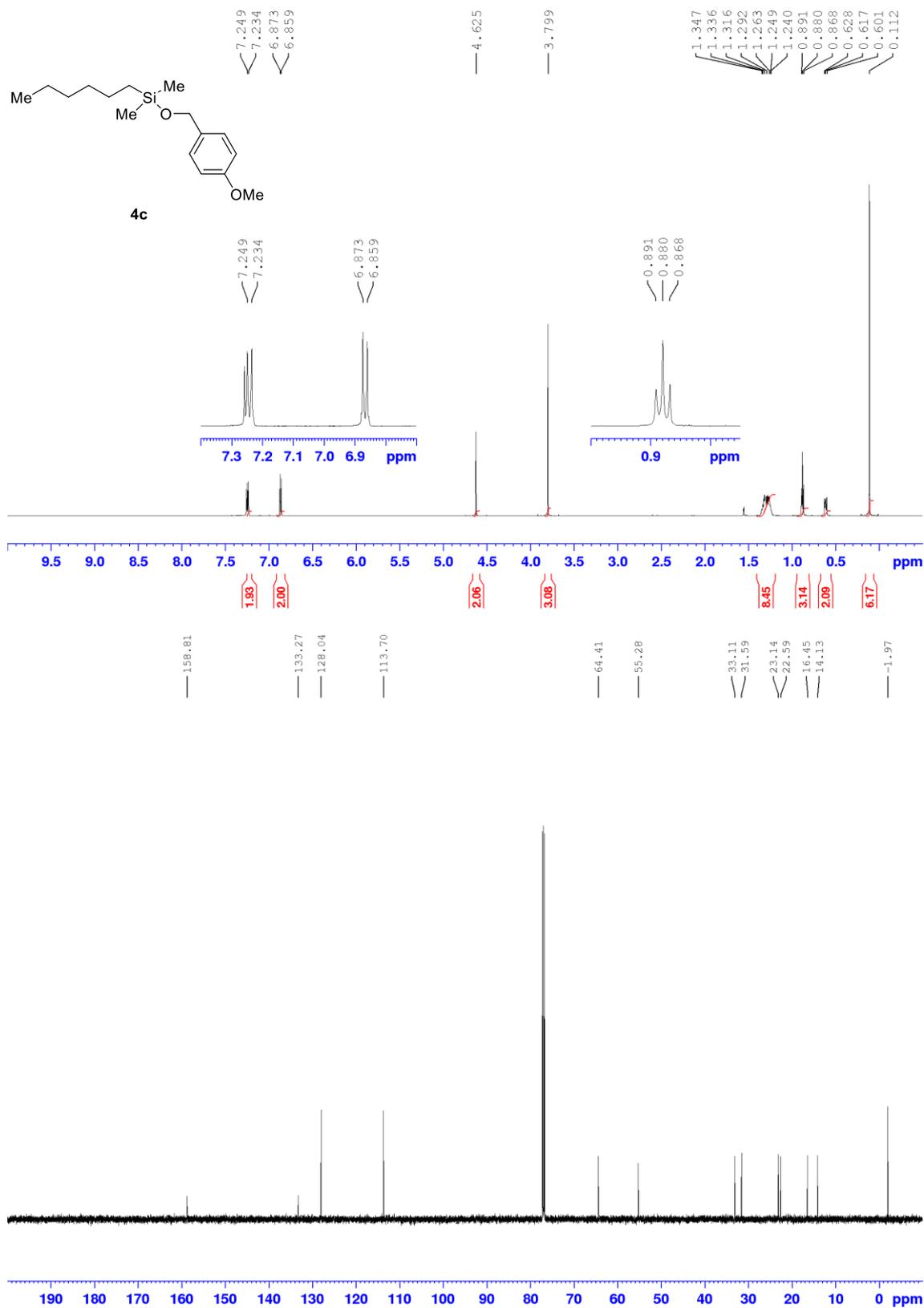
¹H (600 MHz, CDCl₃) and ¹³C NMR Spectra (150 MHz, CDCl₃) of 2p



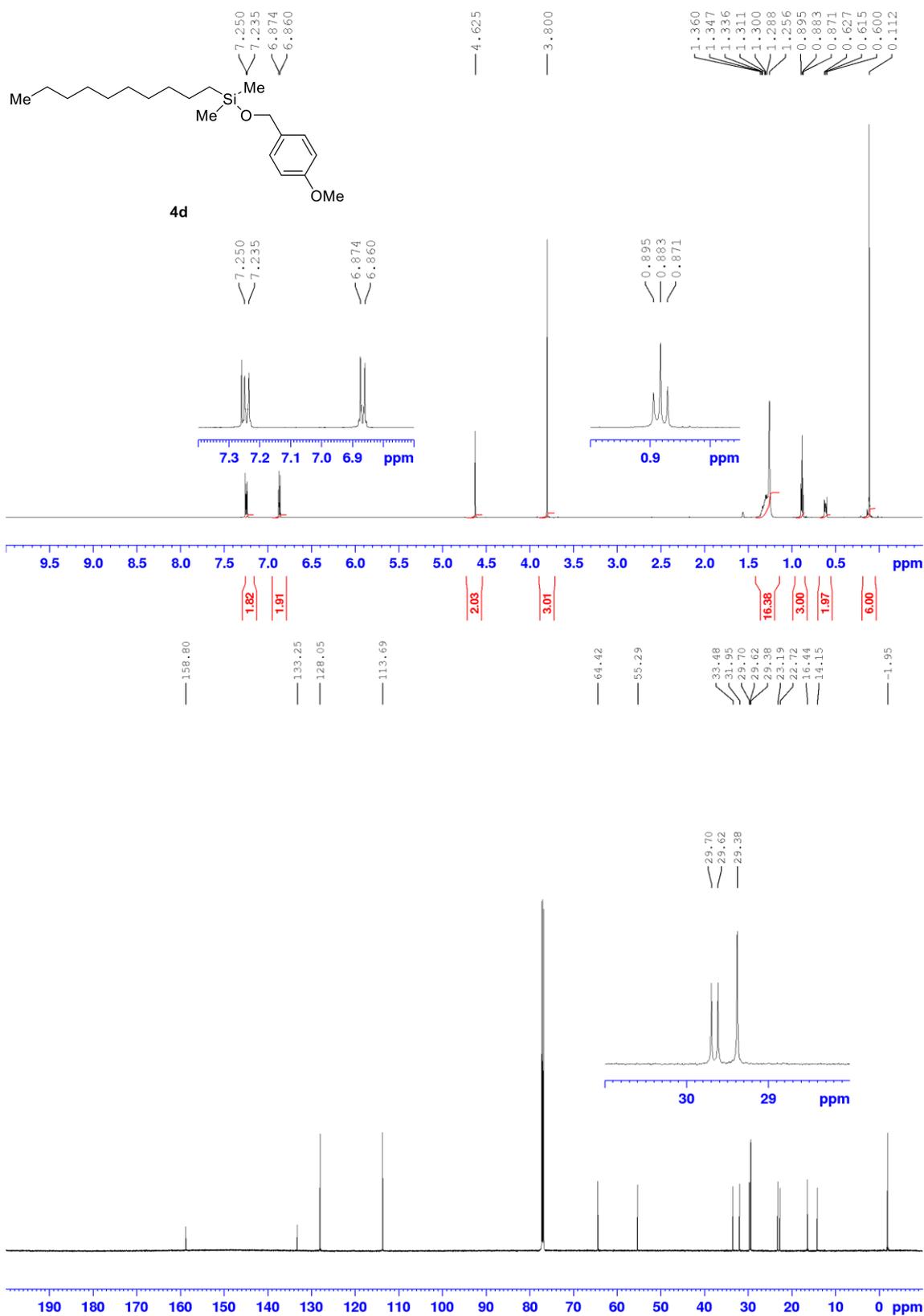
^1H (600 MHz, CDCl_3) and ^{13}C NMR Spectra (150 MHz, CDCl_3) of 4b



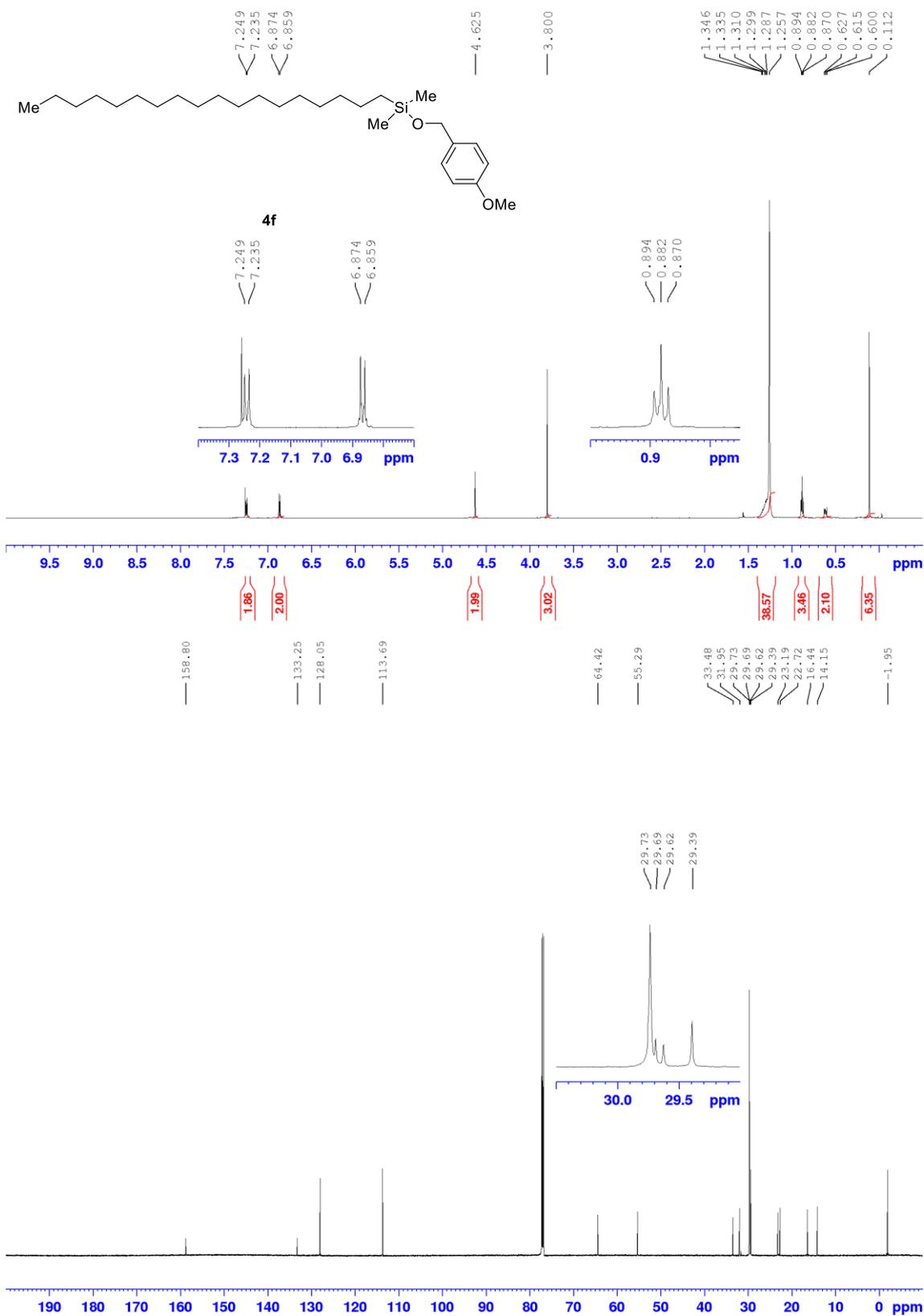
^1H (600 MHz, CDCl_3) and ^{13}C NMR Spectra (150 MHz, CDCl_3) of 4c



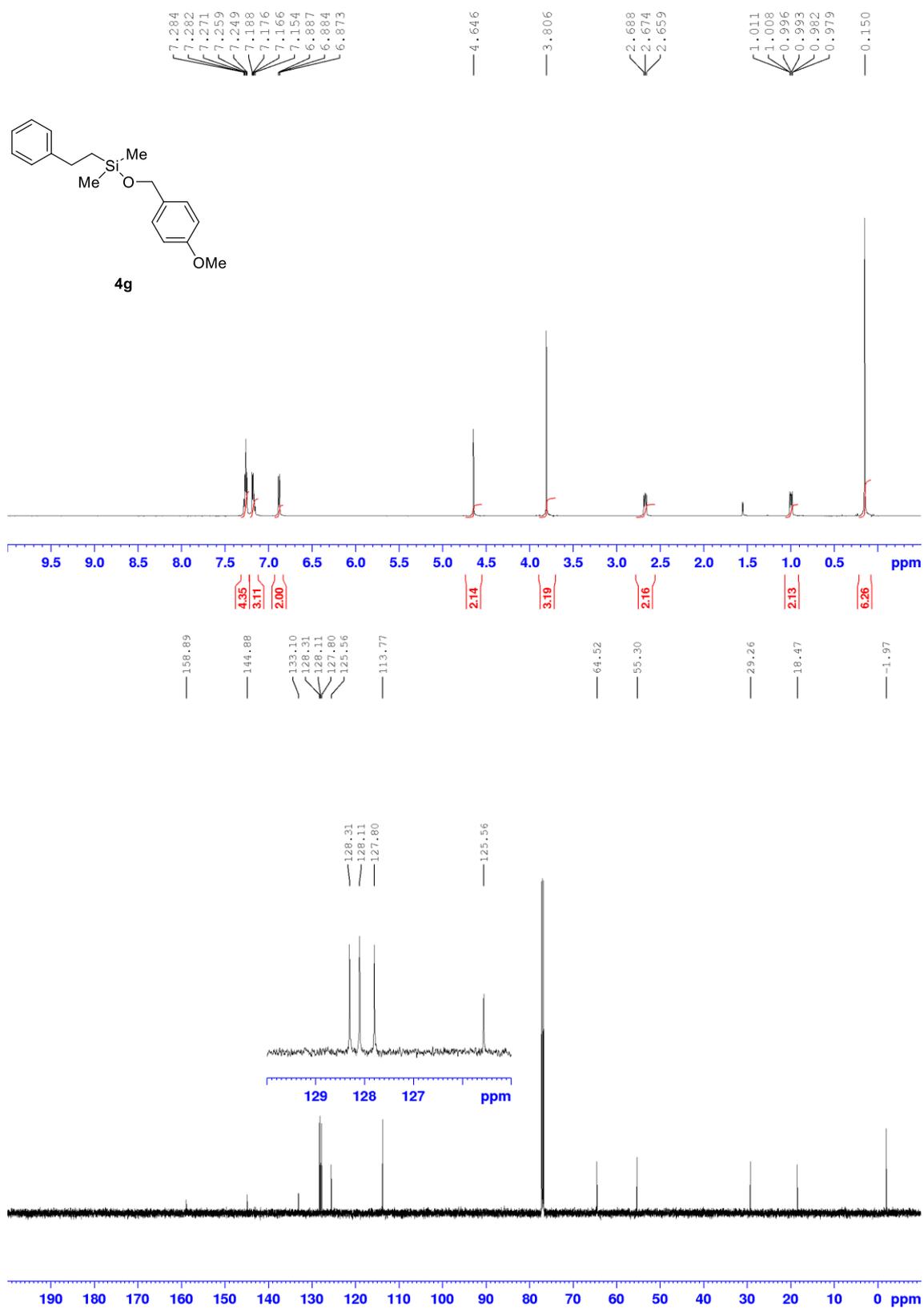
¹H (600 MHz, CDCl₃) and ¹³C NMR Spectra (150 MHz, CDCl₃) of 4d



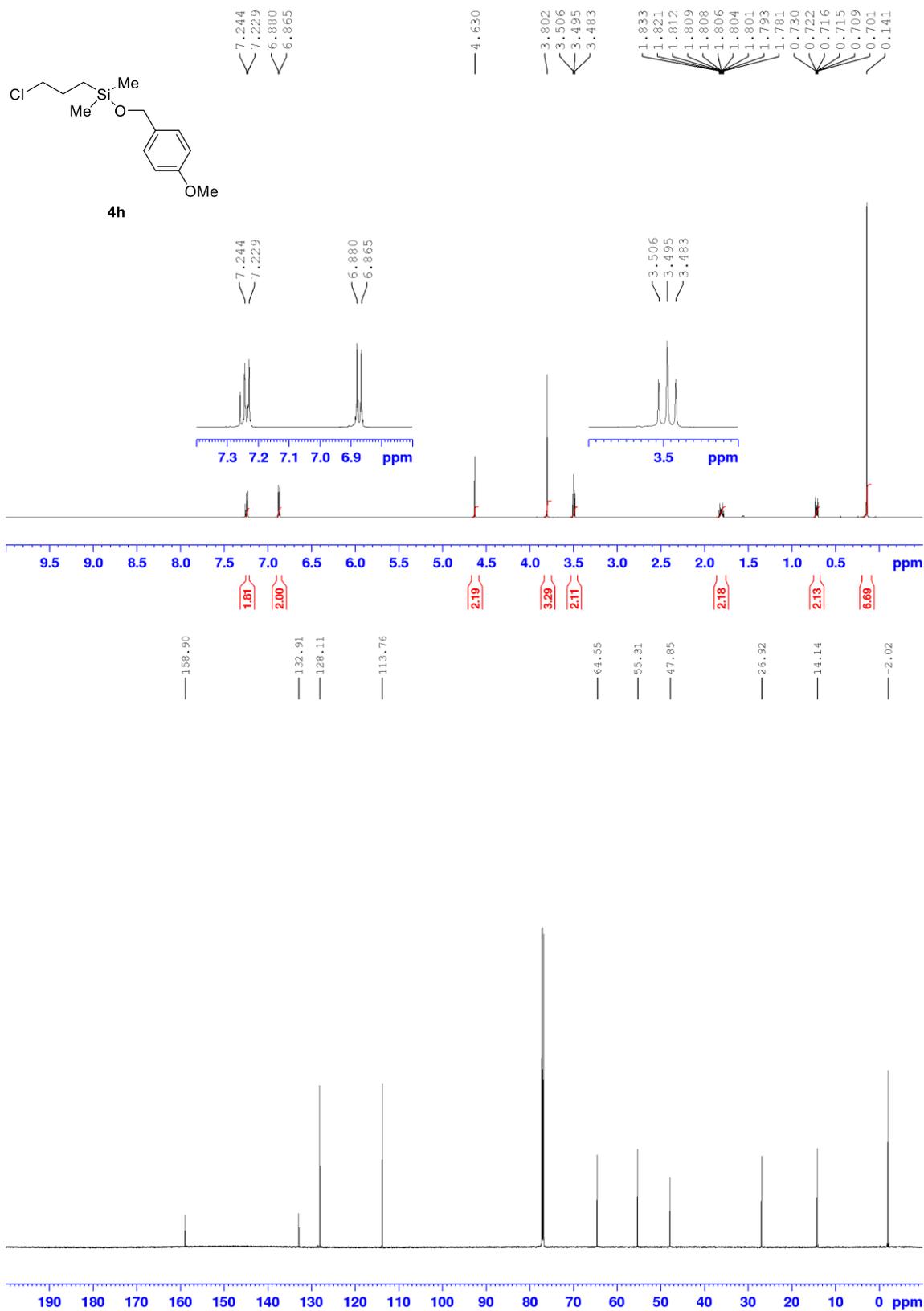
^1H (600 MHz, CDCl_3) and ^{13}C NMR Spectra (150 MHz, CDCl_3) of 4f



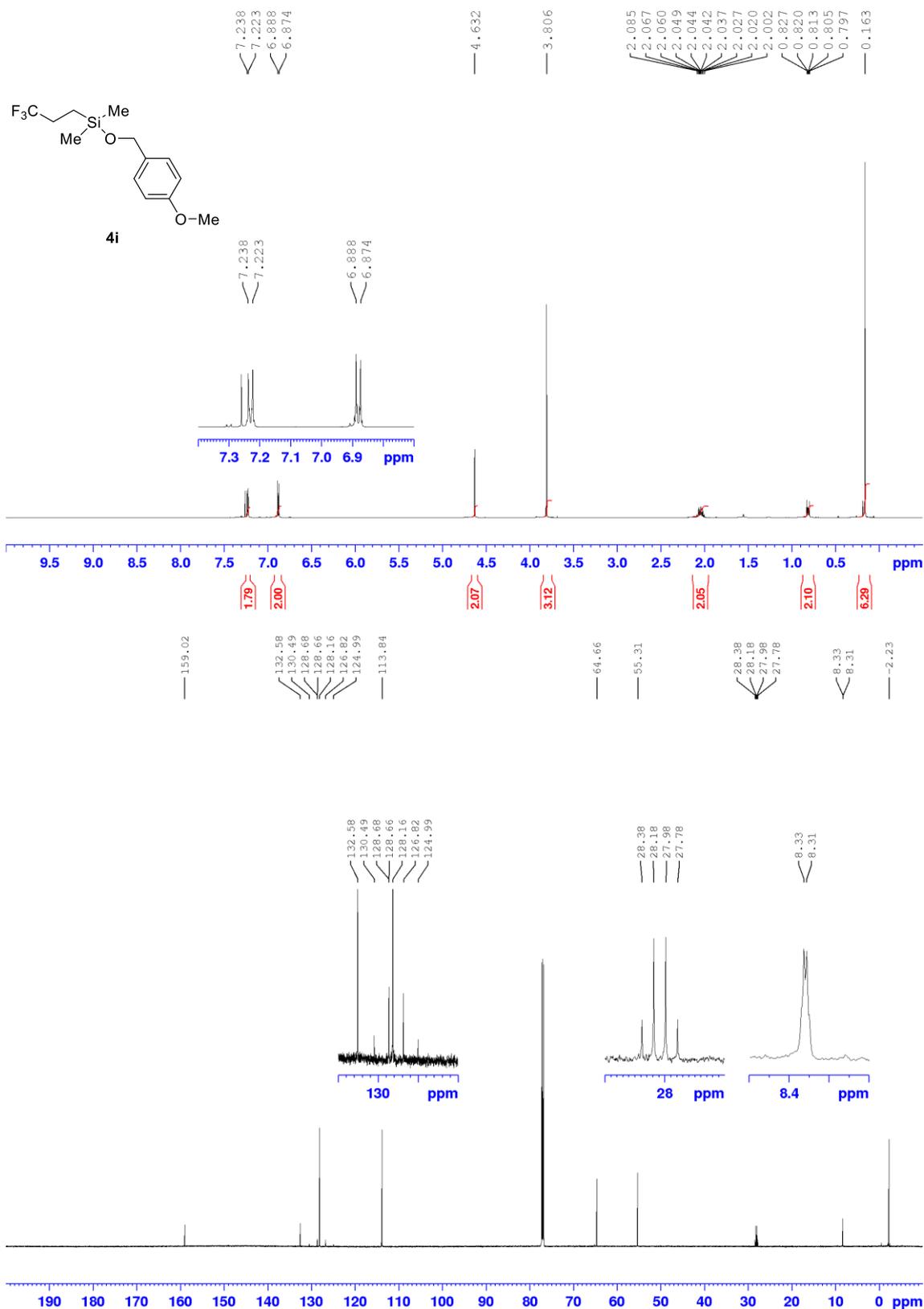
^1H (600 MHz, CDCl_3) and ^{13}C NMR Spectra (150 MHz, CDCl_3) of 4g



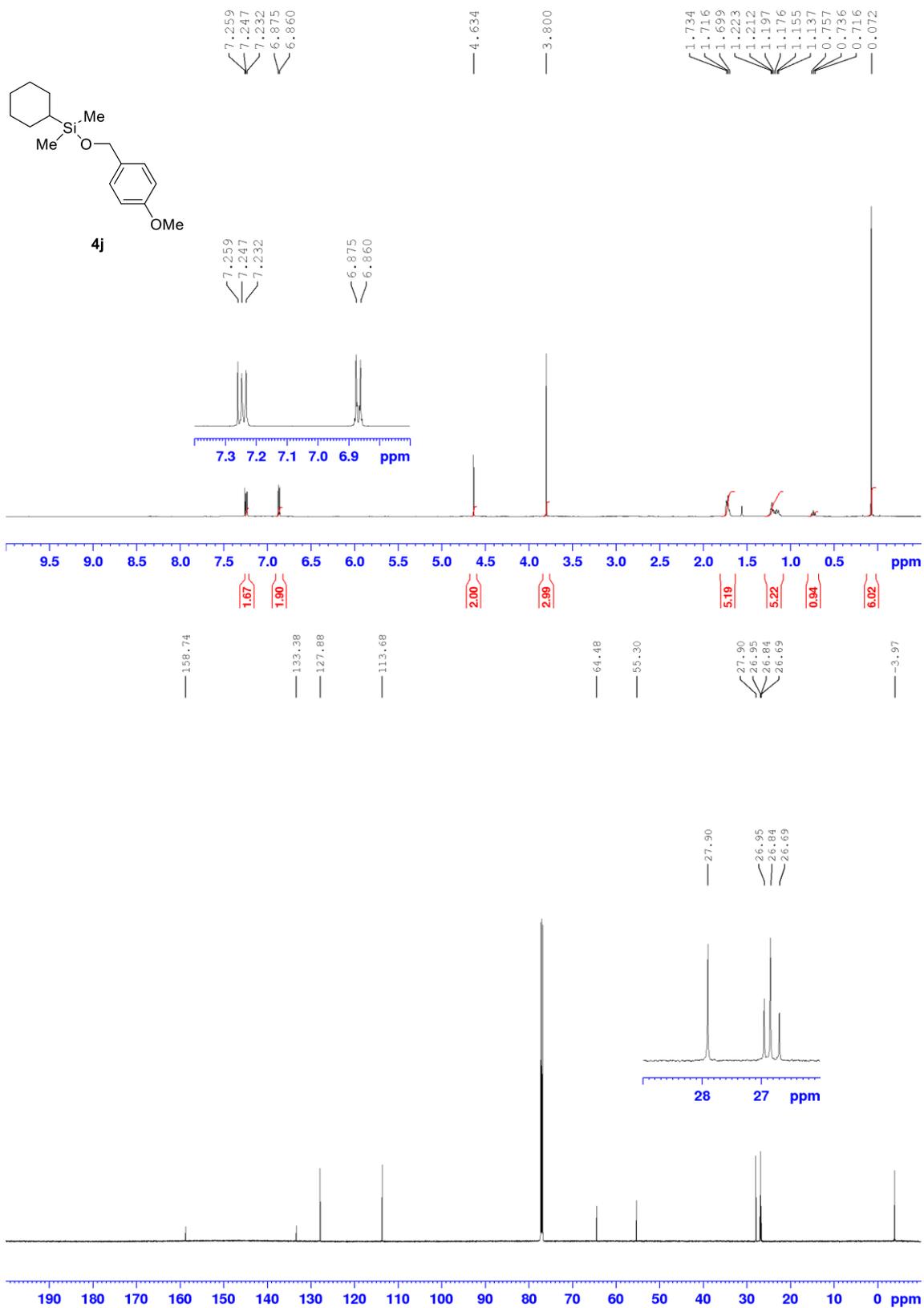
^1H (600 MHz, CDCl_3) and ^{13}C NMR Spectra (150 MHz, CDCl_3) of 4h



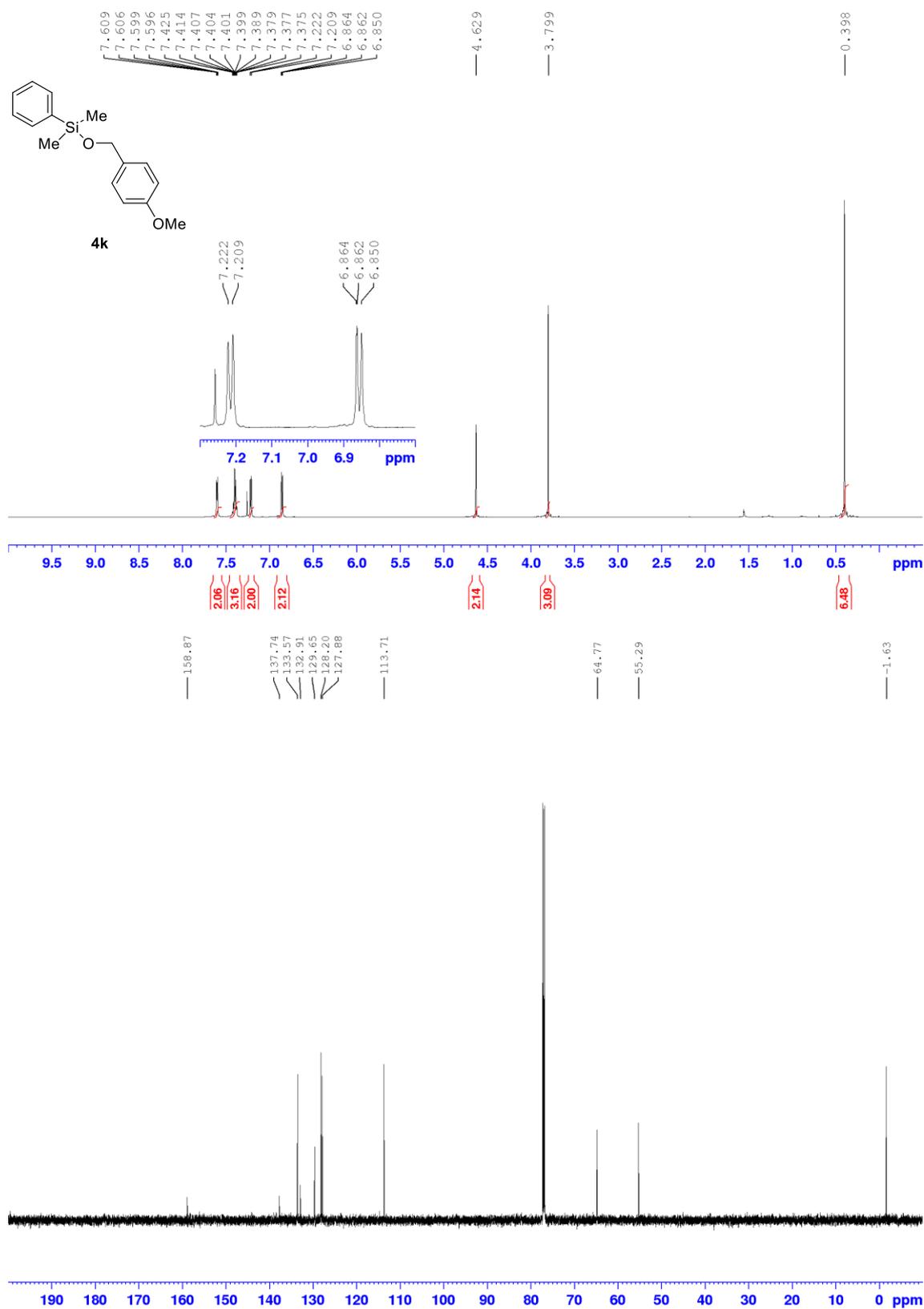
^1H (600 MHz, CDCl_3) and ^{13}C NMR Spectra (150 MHz, CDCl_3) of 4i



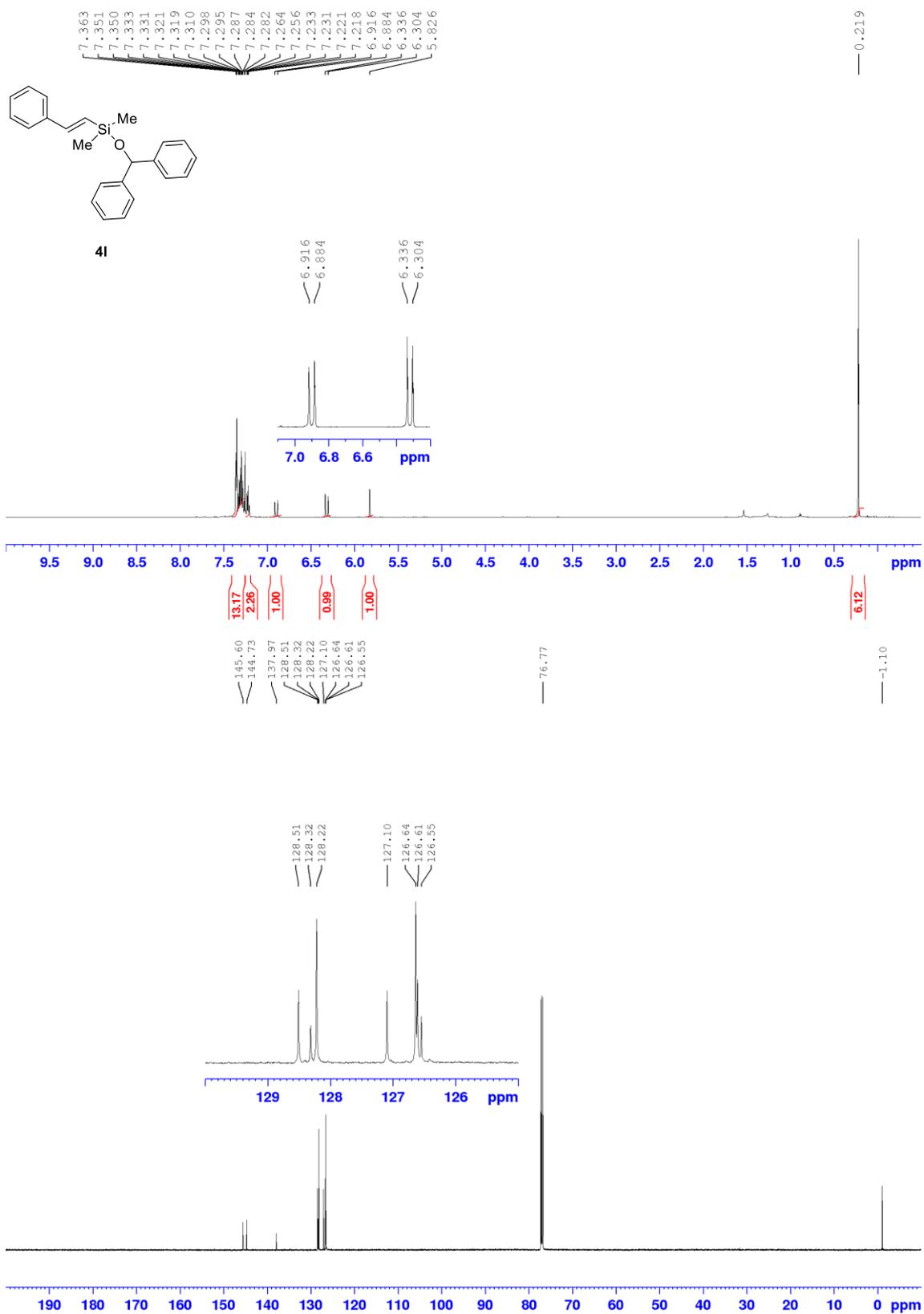
¹H (600 MHz, CDCl₃) and ¹³C NMR Spectra (150 MHz, CDCl₃) of 4j



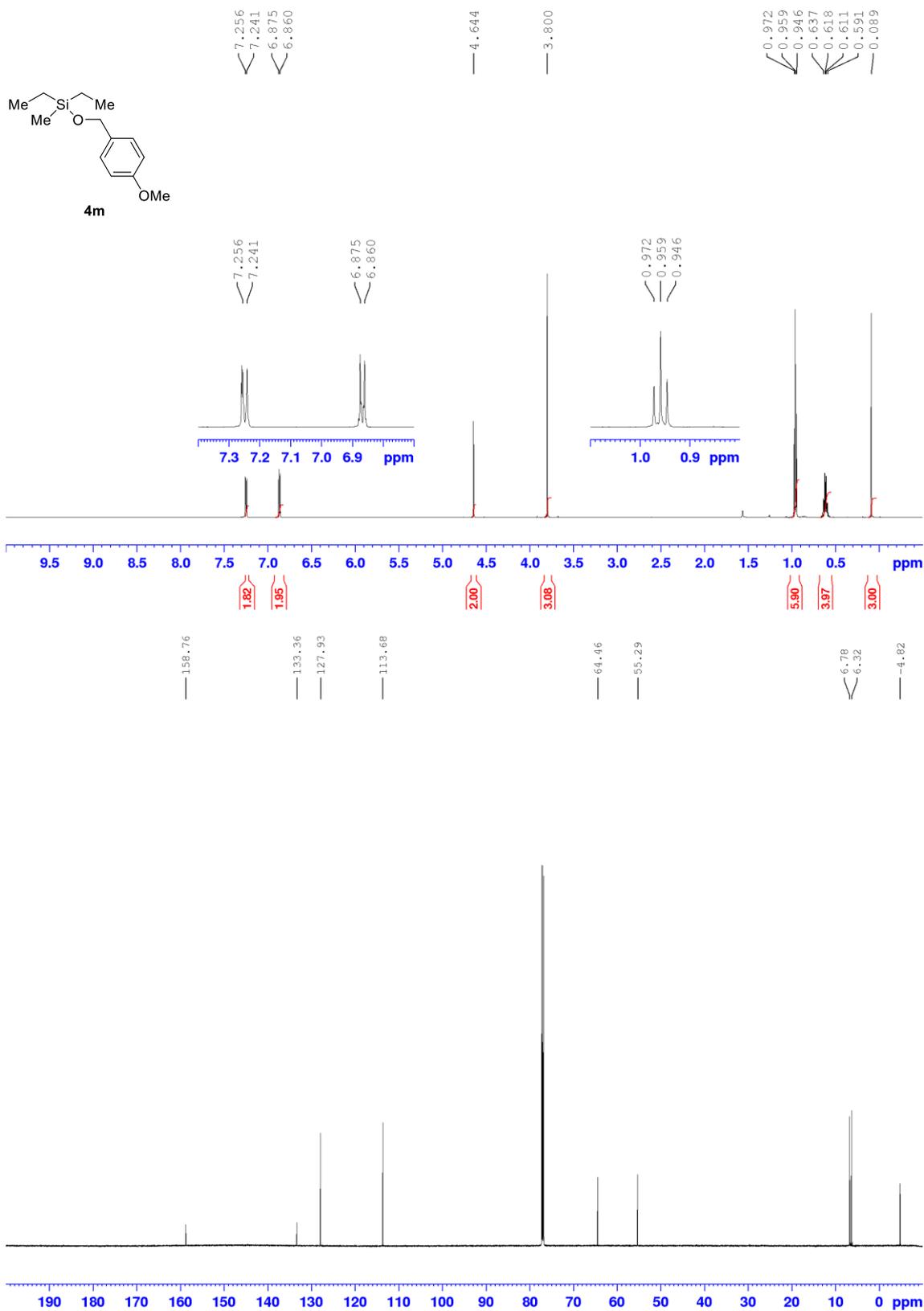
¹H (600 MHz, CDCl₃) and ¹³C NMR Spectra (150 MHz, CDCl₃) of 4k



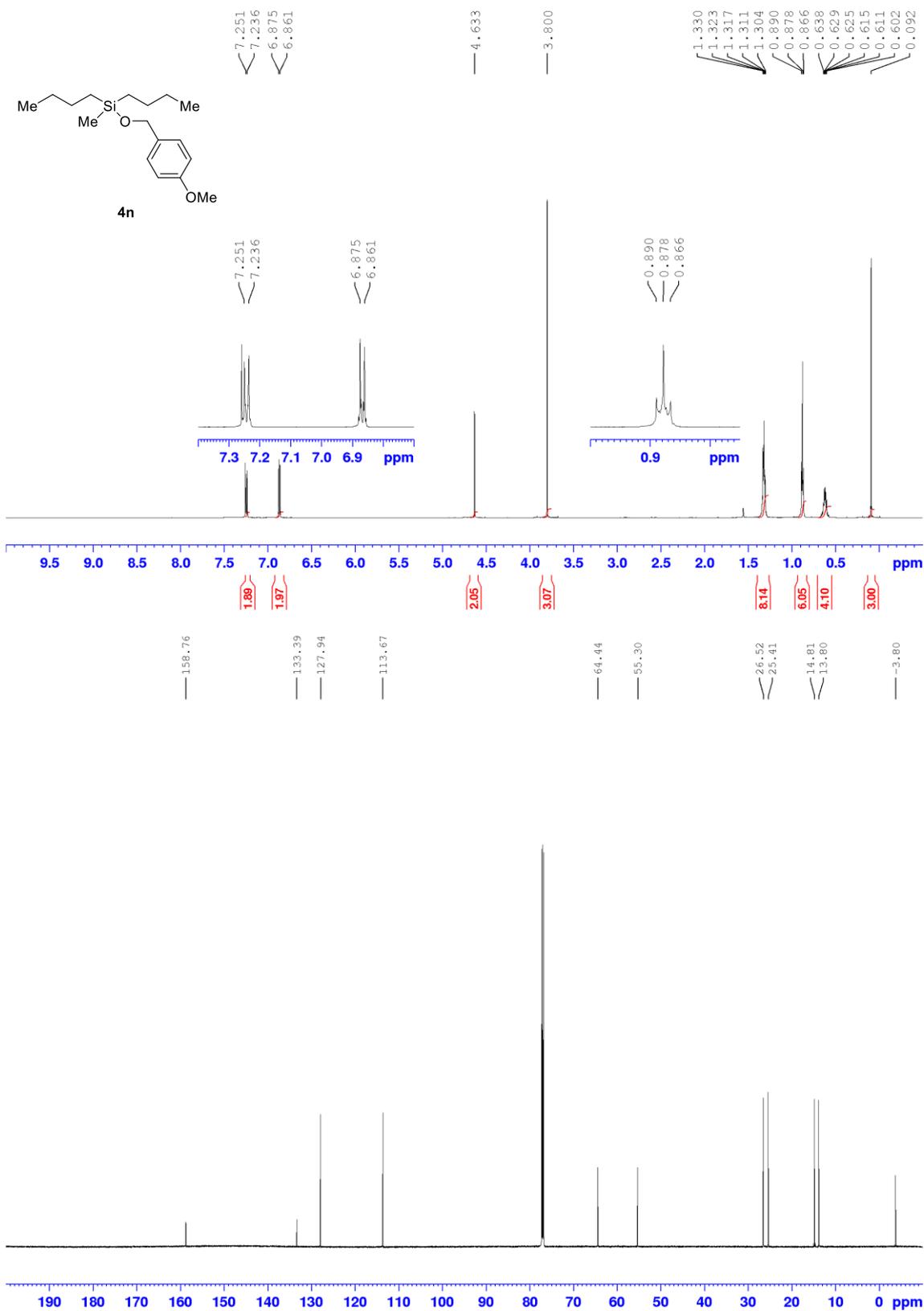
^1H (600 MHz, CDCl_3) and ^{13}C NMR Spectra (150 MHz, CDCl_3) of 4l



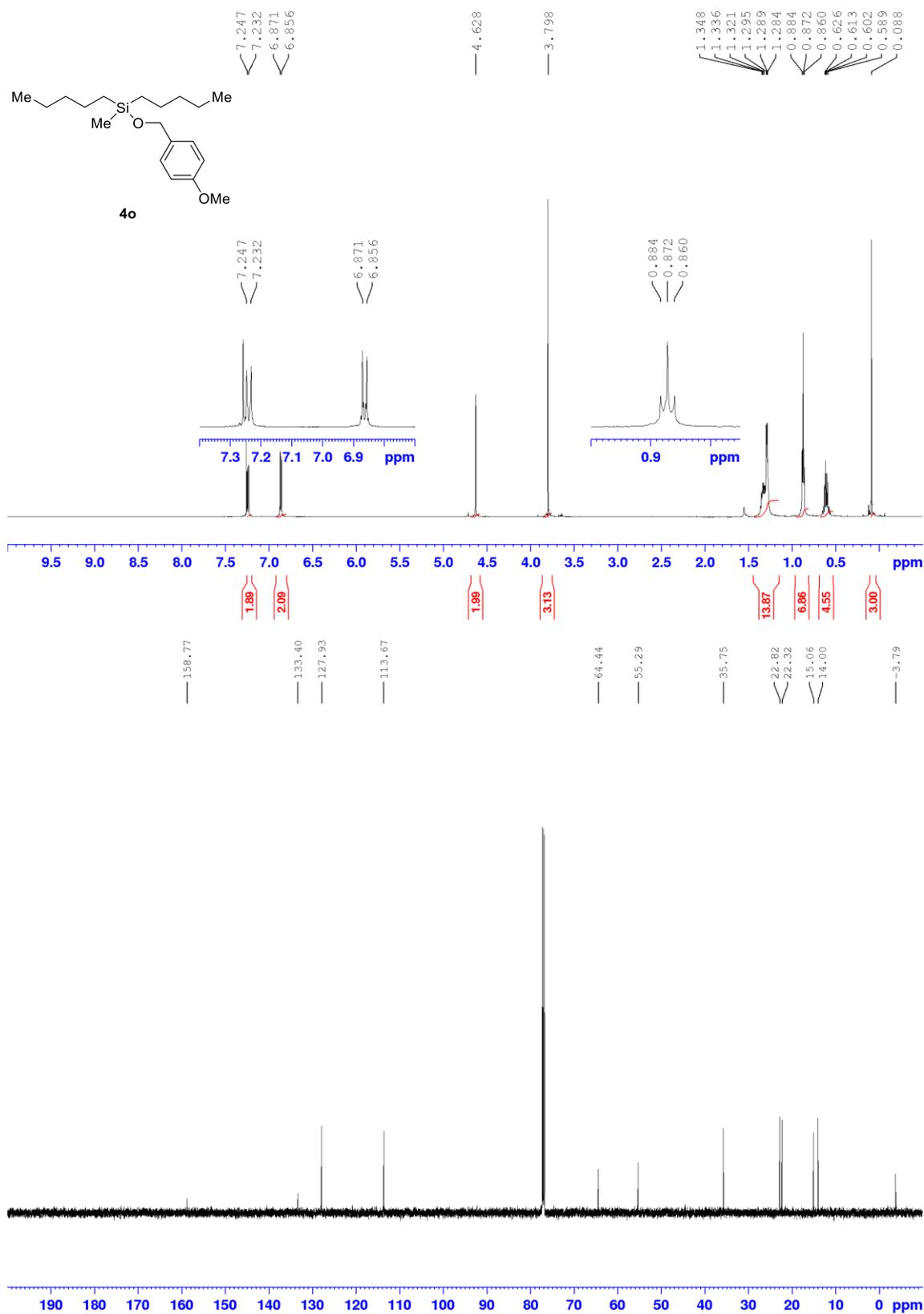
^1H (600 MHz, CDCl_3) and ^{13}C NMR Spectra (150 MHz, CDCl_3) of 4m



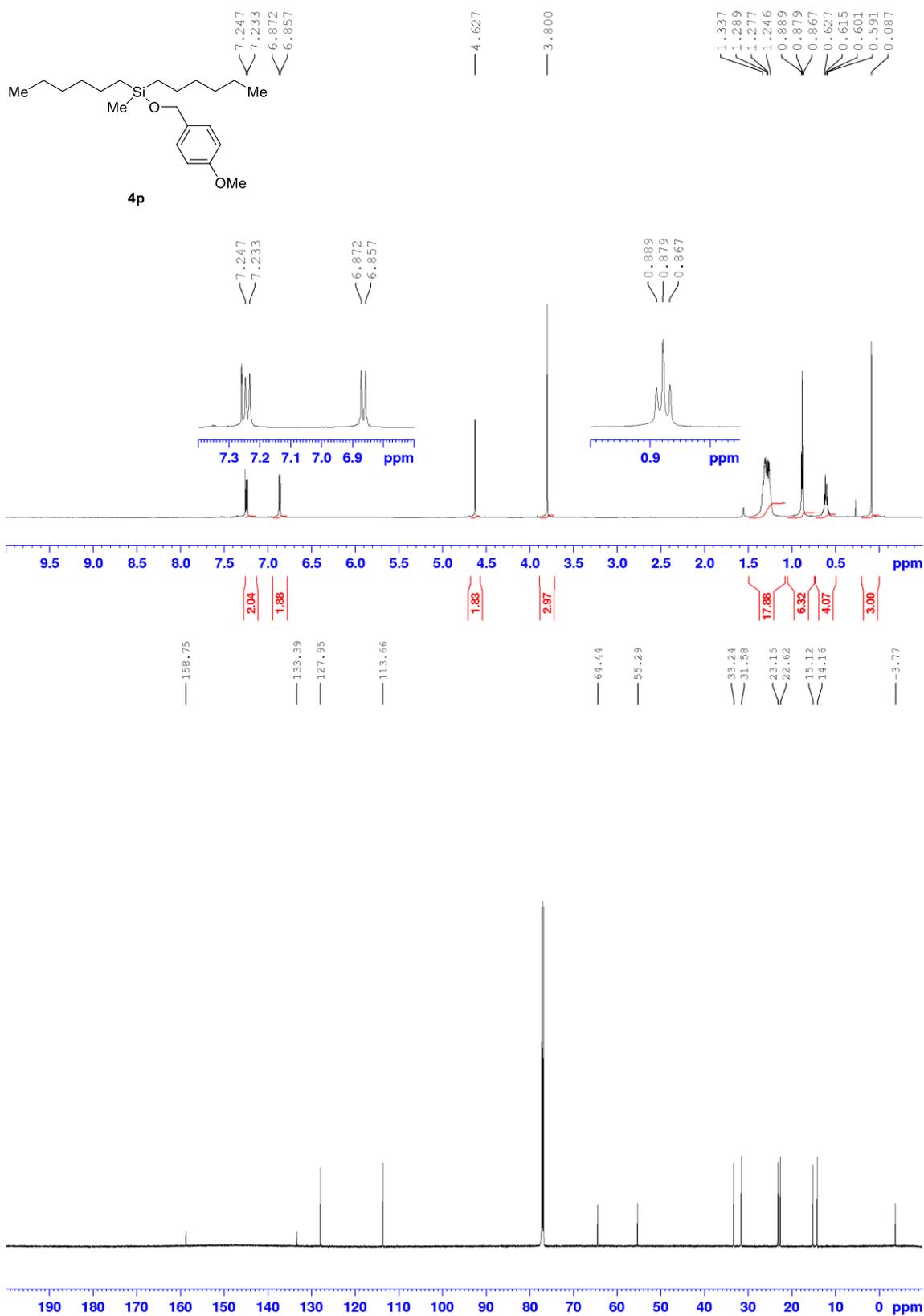
^1H (600 MHz, CDCl_3) and ^{13}C NMR Spectra (150 MHz, CDCl_3) of 4n



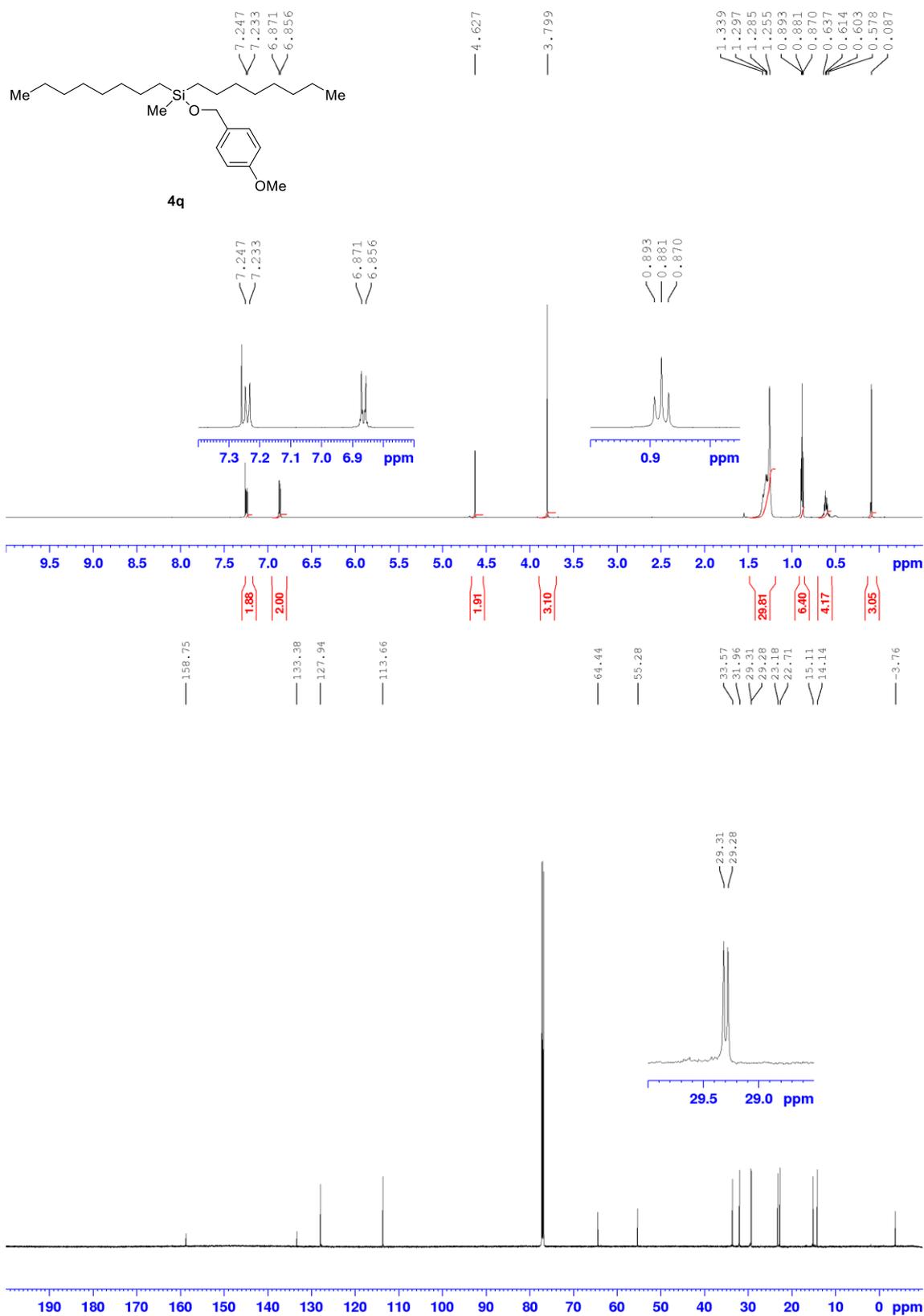
^1H (600 MHz, CDCl_3) and ^{13}C NMR Spectra (150 MHz, CDCl_3) of 4o



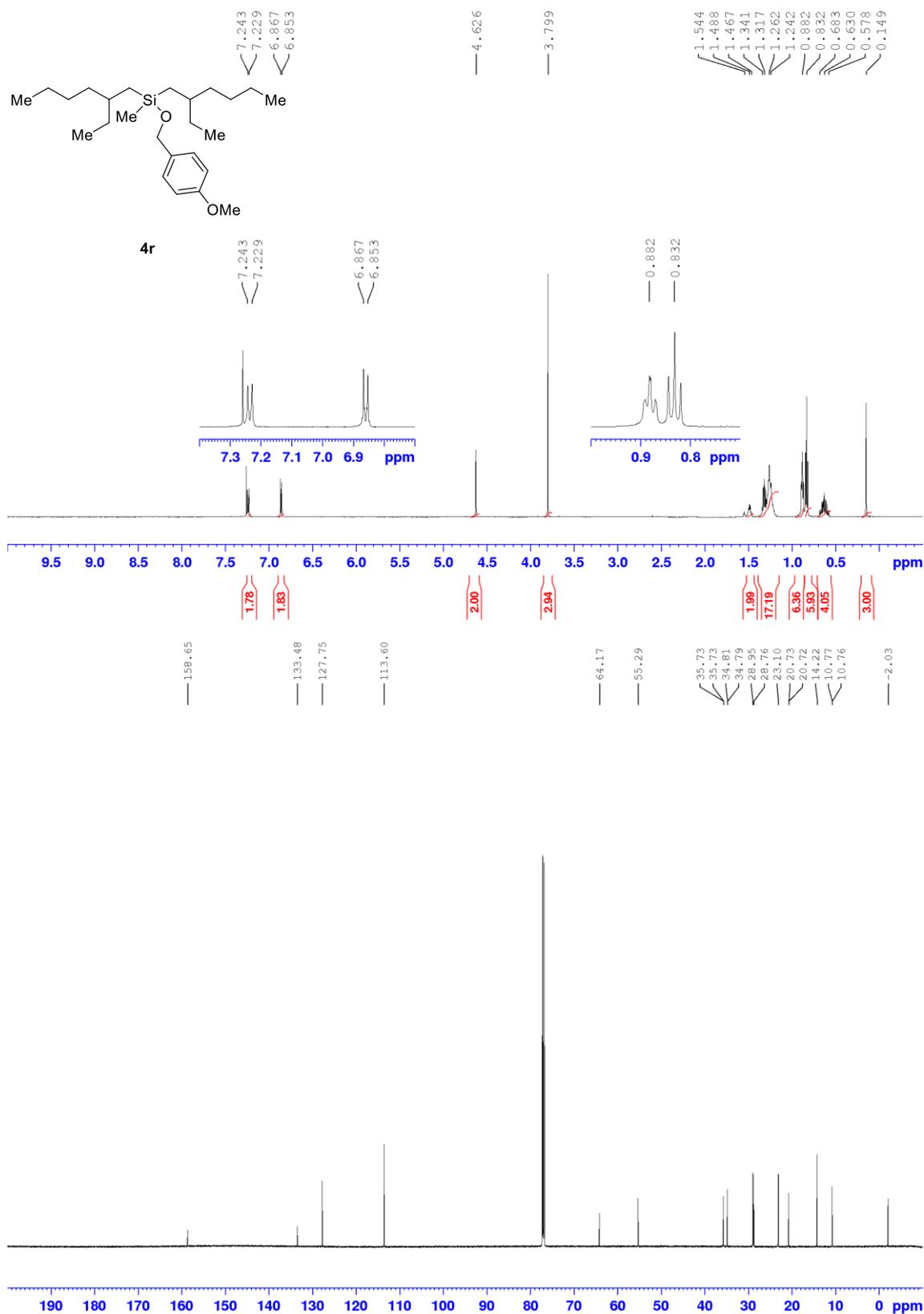
^1H (600 MHz, CDCl_3) and ^{13}C NMR Spectra (150 MHz, CDCl_3) of 4p



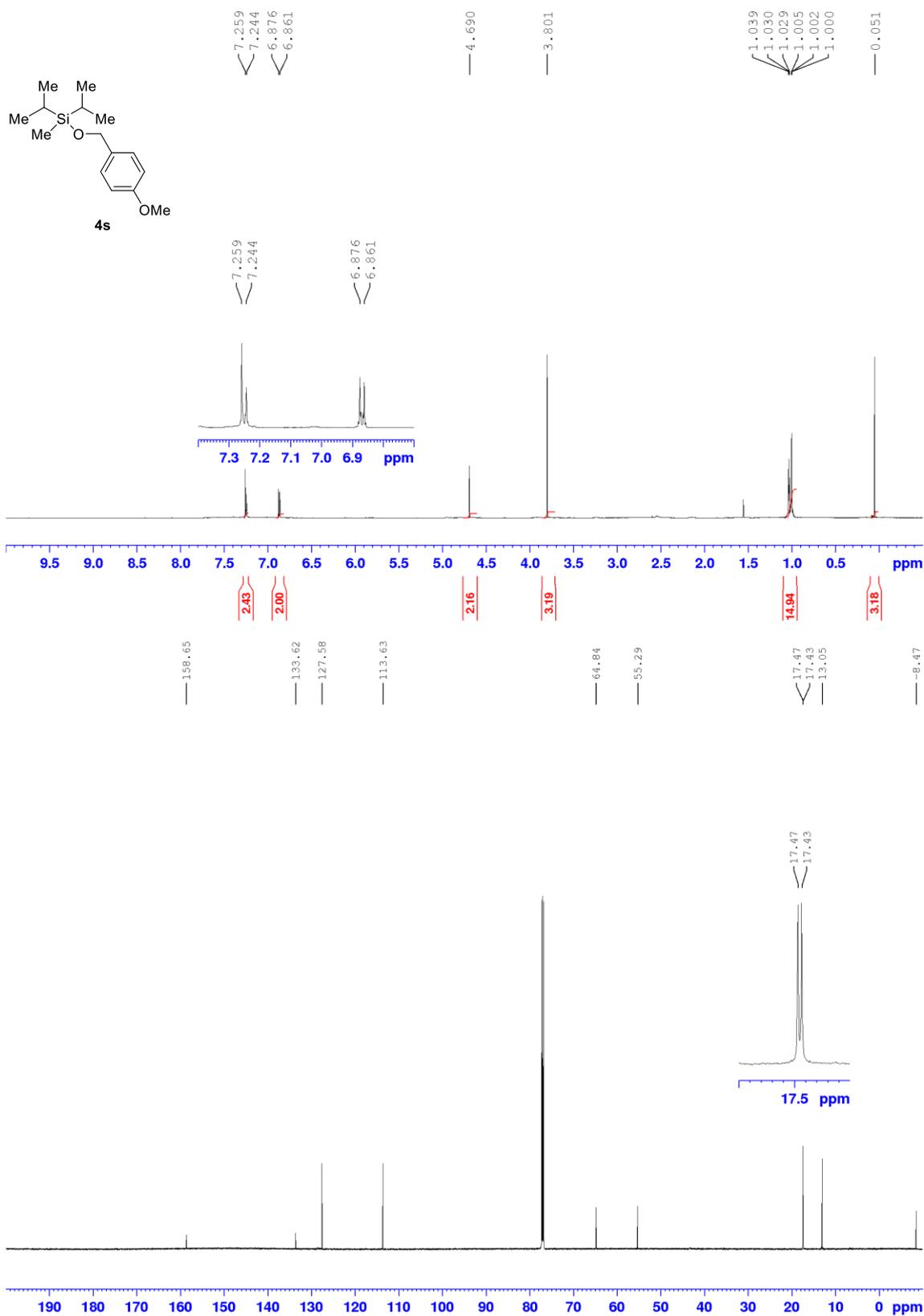
¹H (600 MHz, CDCl₃) and ¹³C NMR Spectra (150 MHz, CDCl₃) of 4q



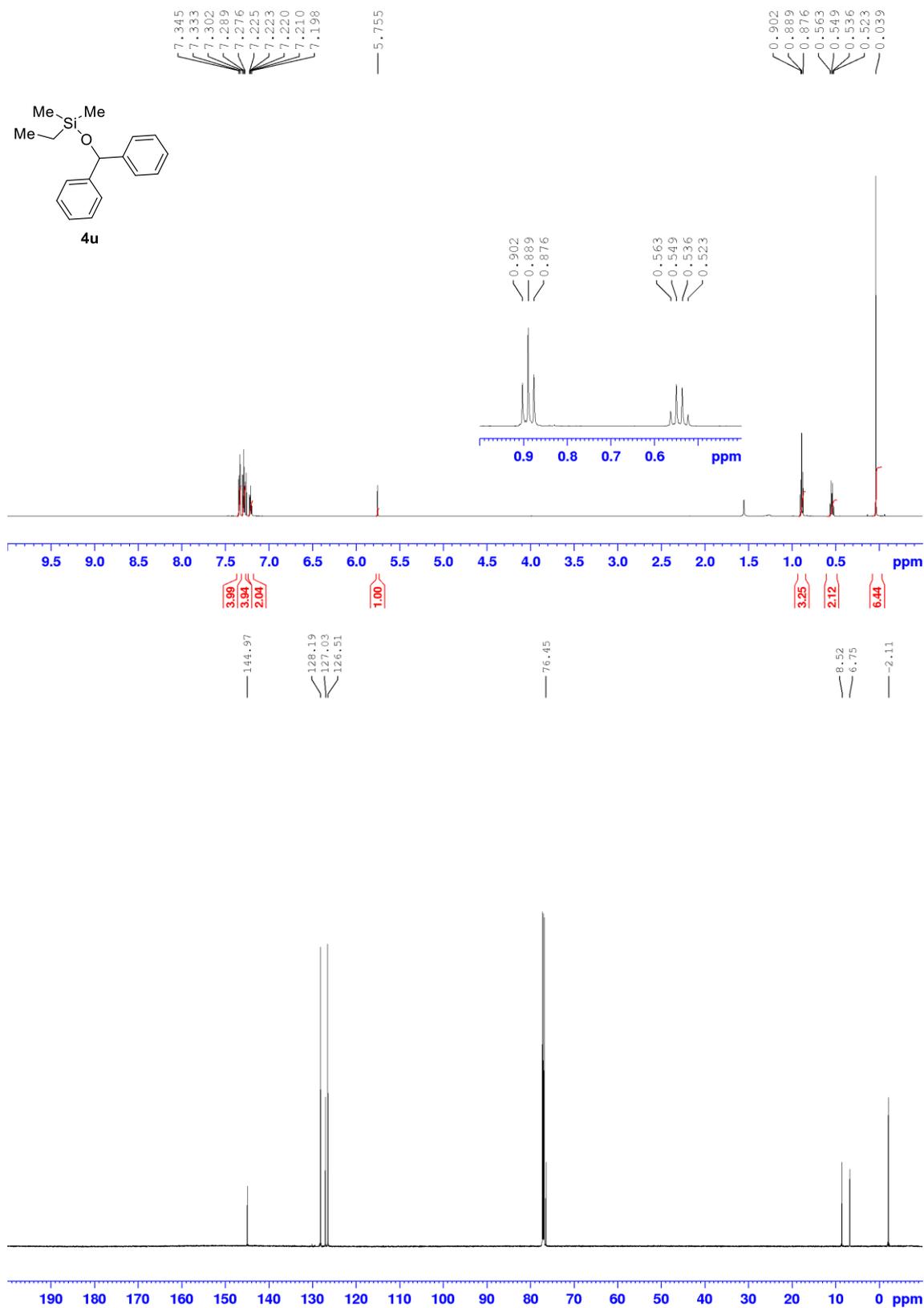
¹H (600 MHz, CDCl₃) and ¹³C NMR Spectra (150 MHz, CDCl₃) of 4r



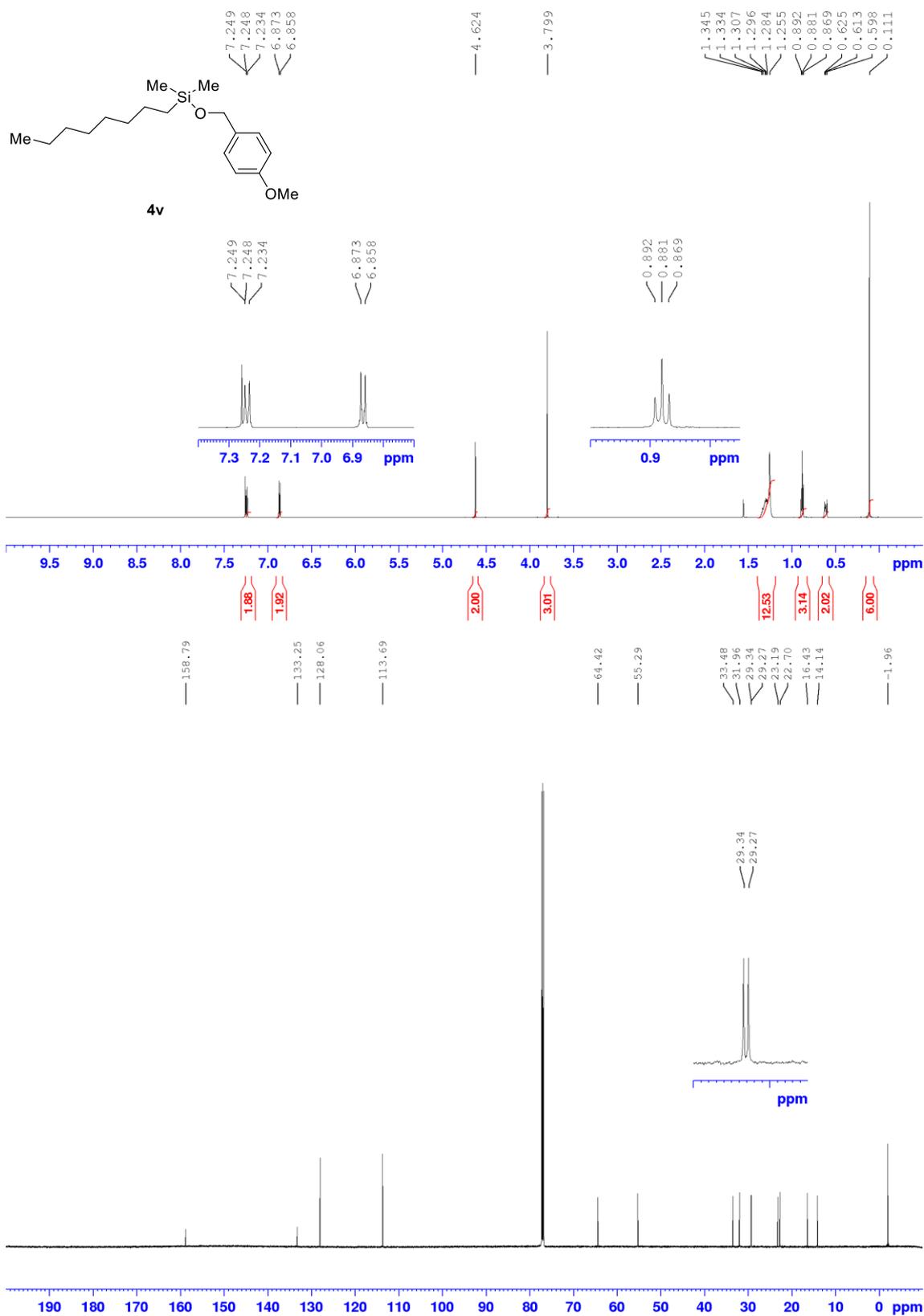
^1H (600 MHz, CDCl_3) and ^{13}C NMR Spectra (150 MHz, CDCl_3) of 4s



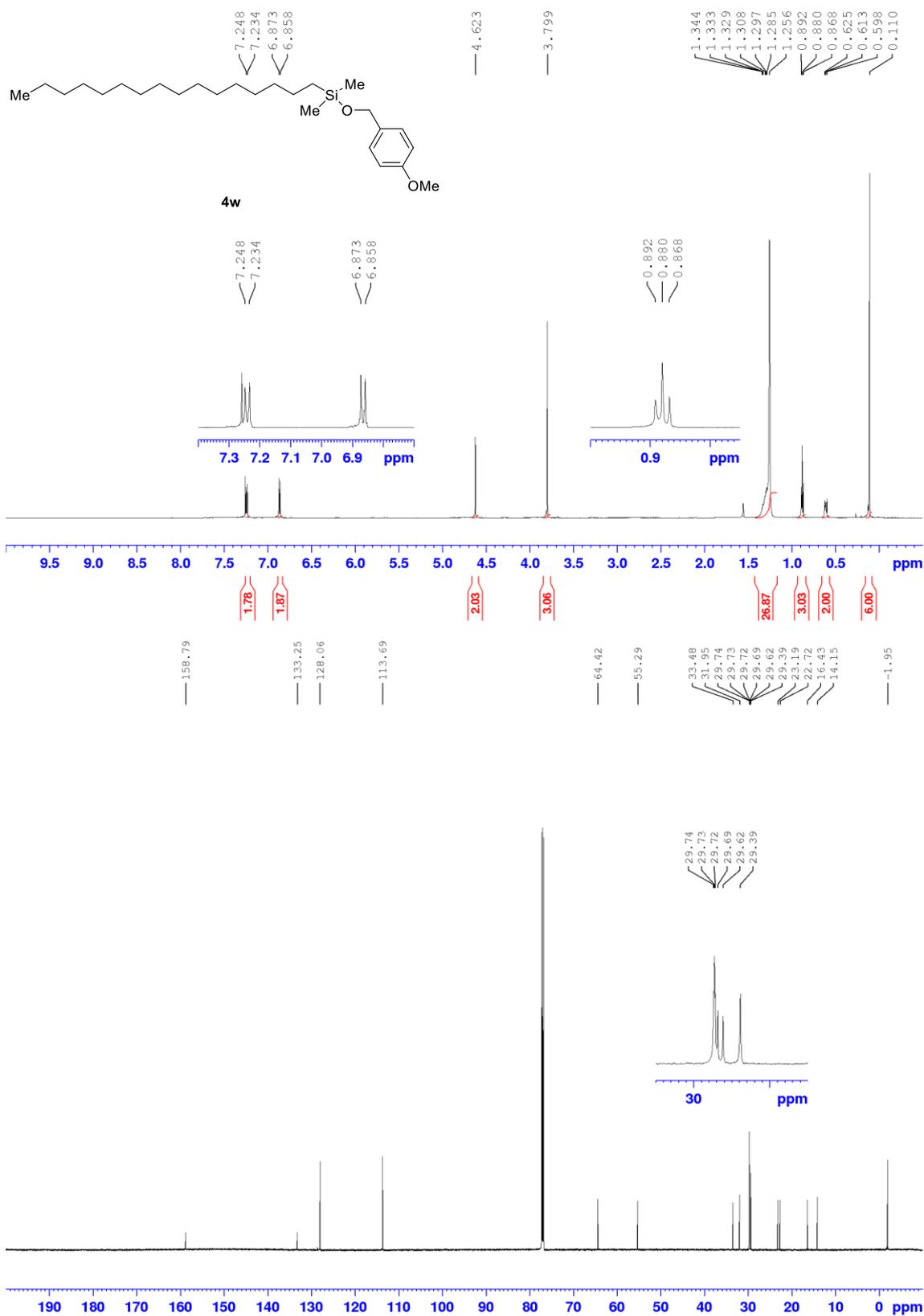
¹H (600 MHz, CDCl₃) and ¹³C NMR Spectra (150 MHz, CDCl₃) of 4u



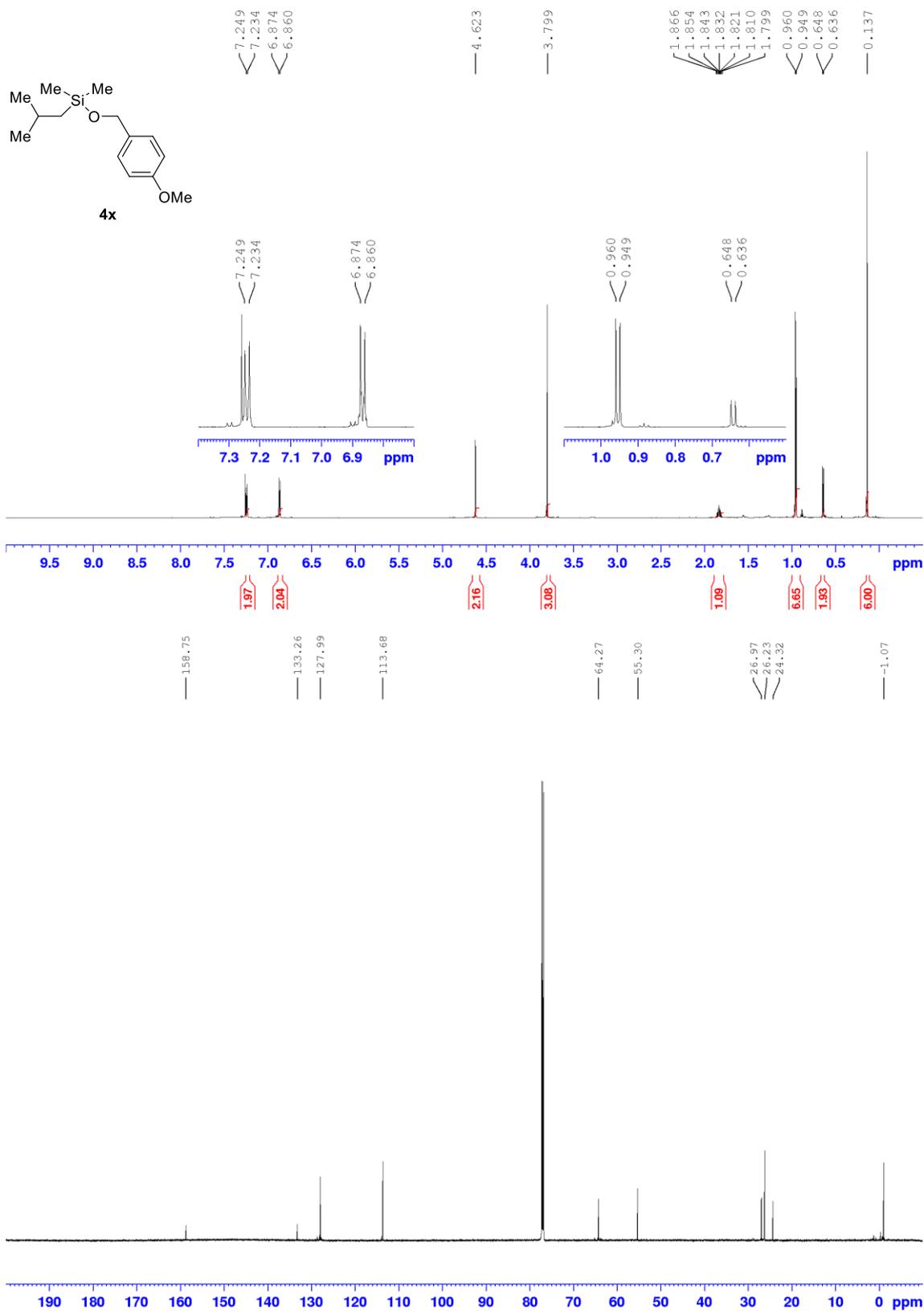
¹H (600 MHz, CDCl₃) and ¹³C NMR Spectra (150 MHz, CDCl₃) of 4v



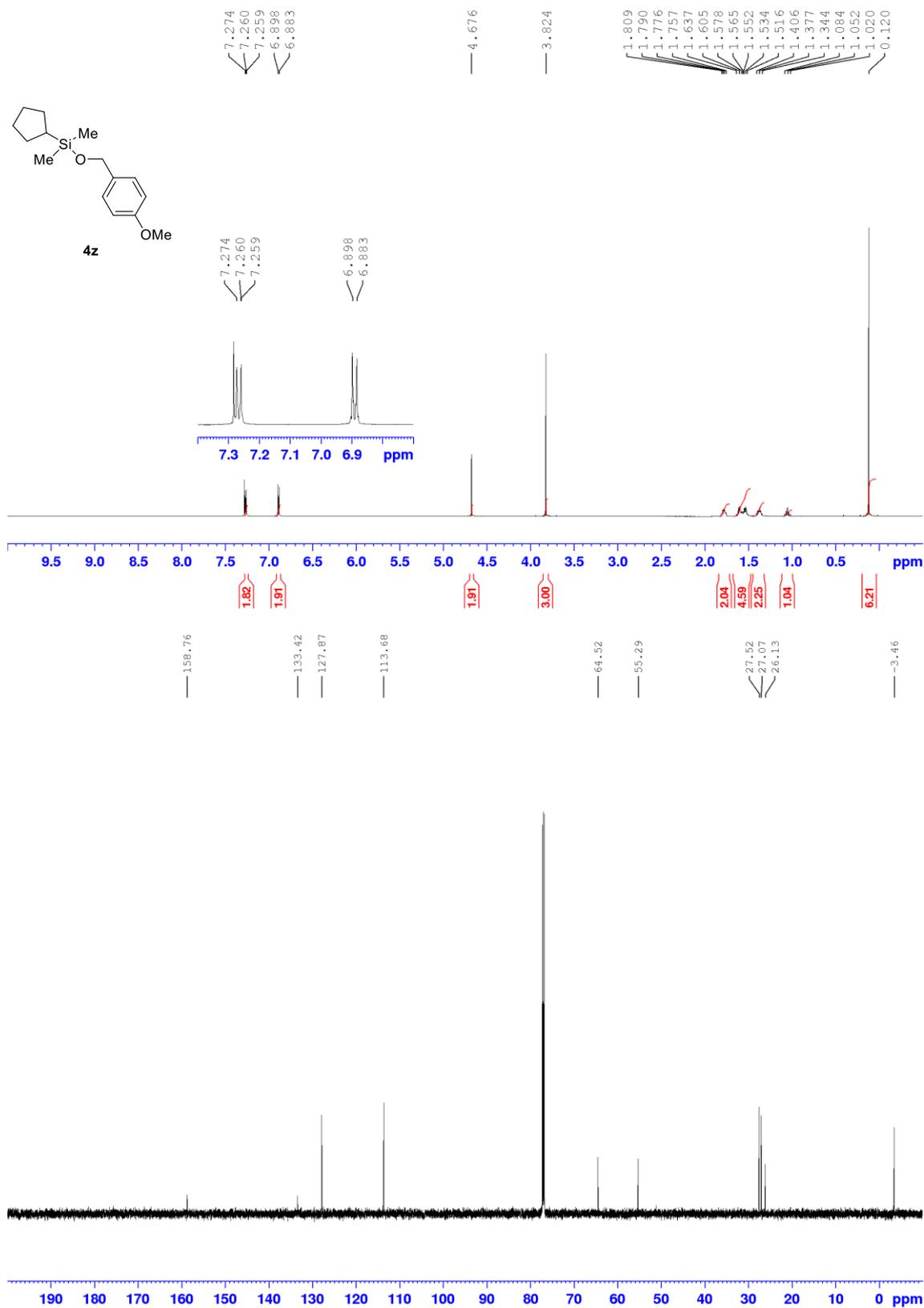
¹H (600 MHz, CDCl₃) and ¹³C NMR Spectra (150 MHz, CDCl₃) of 4w



¹H (600 MHz, CDCl₃) and ¹³C NMR Spectra (150 MHz, CDCl₃) of 4x



^1H (600 MHz, CDCl_3) and ^{13}C NMR Spectra (150 MHz, CDCl_3) of 4z



^1H (600 MHz, CDCl_3) and ^{13}C NMR Spectra (150 MHz, CDCl_3) of 4aa

