SUPPORTING INFORMATION

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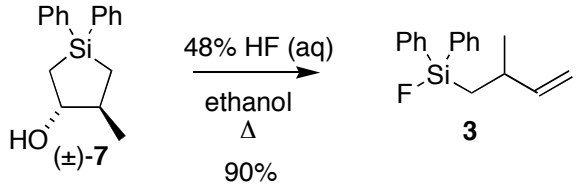
experimental – ^{1}H spectrum



Fluoro (2-methylbut-3-enyl)-diphenylsilane (3). Before running this reaction, practitioners should familiarize themselves with the insidious burns that can result from HF exposure.[1] To a solution of alcohol **7** (6 g, 22.4 mmol) in ethanol (50 mL) was added 48% hydrofluoric acid (15 mL) and the reaction mixture was heated to reflux for 8 h. The mixture was extracted with hexanes (5 x 30mL). The combined organics were washed with water (10 mL), brine (10 mL) and then dried over $MgSO_4$. Concentration and kugelrohr distillation (130 – 140 °C, 5 mm Hg) gave fluorosilane **3** as a colorless oil (5.44 g, 90%).

R_f = 0.6 (hexanes)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72-7.69 (m, 4H), 7.52-7.45 (m, 6H), 5.80 (ddt, J =17.4, 10.2, 7.2 Hz, 1H), 4.93 (dd, J = 18.4, 13.2 Hz, 2H), 2.59 (m, 1H), 1.38 (m, 1H), 1.30 (m, 1H), 1.17 (d, J = 6.8 Hz, 3H).

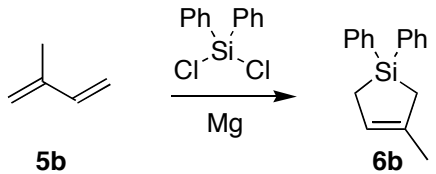
^{13}C NMR (100 MHz, $CDCl_3$) δ 145.7, 134.0, 130.4, 128.0. 111.5, 32.8, 23.0, 22.3 (d, J_{CF} = 13.5 Hz).

^{19}F NMR (282.2 MHz, CDCl3) δ -169.5.

IR (neat) cm^{-1} 3072, 2960, 1429, 1124, 847.

Exact mass (FAB) $(M + Na)^+$ calc. $C_{17}H_{19}FSi$ 270.1240 found 270.1232.

[1] Blodgett. D. W.; Suruda, A. J.; Crouch, B. I. *Am. J. Indust. Med.* **2001**, *40*, 215-220.



5b **6b**

2,5-Dihydro-3-methyl-1,1-diphenylsilole (6b). Following the method of Mignani,[2] a suspension of magnesium powder (50 mesh, 1.91g, 78.9 g-atom) in THF (60 mL) under nitrogen was added dropwise diphenyldichlorosilane (16.3 mL, 78.9 mmol), followed by isoprene (15.8 mL, 158 mmol) and then phenyl magnesium chloride (2M in THF, 2 mL, 4 mmol). After vigorous stirring for 1 h, the mixture was irradiated with a sun lamp for 2 h. After stirring for an additional 6 days or until the magnesium has been consumed, the mixture was cooled to 0 °C and water was added cautiously. After stirring for an additional hour, the aqueous phase was extracted with hexanes (5 x 30 mL). The combined organics were dried over $MgSO_4$ and concentrated. Kugelrohr distillation (110 – 120 °C/ 0.7 mm, Lit. 147 °C/0.25 mm[3]) gave **6b** as a colorless oil (14.6 g, 74%).

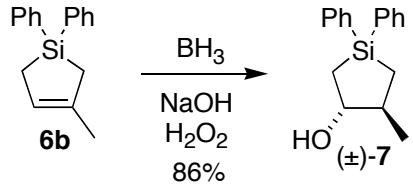
R_f = 0.51 (hexanes).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76-7.74 (m, 4H), 7.56-7.50 (m, 6H), 5.64 (m, 1H), 1.84 (s, 2H & 3H), 1.78 (s, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 139.1, 135.12, 133.65, 128.4, 126.8, 123.7, 21.5, 20.7, 16.5.

IR (neat) 3067, 3001, 2909, 2882, 1428, 1114 cm^{-1}.

[2] Mignani, S., Damour, D., Bastart, J., Manuel, G. *Synth. Commun.* **1995,** *25,* 3855-61.
[3] Manuel, G., Mazerolles, P., Lesbre, M. Pradel, J.-P. *J. Organomet. Chem.* **1973**, *61*, 147-165.



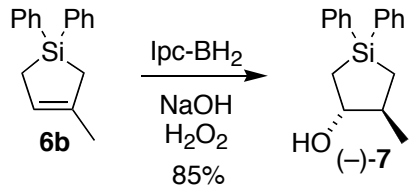
Racemic *trans*-4-Methyl-3-hydroxy-1,1-diphenylsilole (±-7). To a solution of **6b** (7.05 g, 28.2 mmol) in THF (40 mL) at 0 °C was added dropwise borane-tetrahydrofuran complex (1M in THF, 28.2 mL, 28.2 mmol). After stirring overnight at ambient temperature, NaOH (9.4 mL, 3N) was added carefully, followed by 30% H_2O_2 (9 mL). The mixture was heated to 50 °C for 2 h, cooled and the aqueous phase was then extracted with ether (4 x 20 mL). The combined organics were washed with saturated aqueous NaCl (5 mL), dried over $MgSO_4$ and concentrated. Flash chromatography (1:9 ethyl acetate/hexanes) gave alcohol **7** as a gummy liquid (6.5 g, 86%).

R_f = 0.28 (1:9 ethyl acetate/hexanes)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60-7.56 (m, 4H), 7.43-7.39 (m, 6H), 3.84 (m, 1H), 1.98 (m, 1H), 1.74 (dd, J = 14.4, 6.4 Hz), 1.60 (s, 1H), 1.50 (dd, J = 15.2, 6.8 Hz, 2H), 1.18 (d, J = 6.4 Hz, 3H), 0.88 (dd, J = 15.2, 10.8 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 136.0, 135.9, 134.7, 134.6, 129.4, 127.9, 79.5, 42.3, 22.5, 20.5, 18.5.

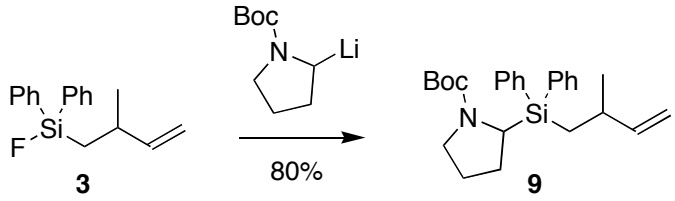
IR (neat) 3375, 3067, 2951, 1428, 1113 cm^{-1}.



(3*S*,4*S*)-*trans*-4-Methyl-3-hydroxy-1,1-diphenylsilole ((–)-7). Monoisopinocamphenyl borane was prepared by a modification of Brown's procedure.[4] To a solution of borane-dimethylsulfide (5.17 mL, 50 mmol) in ether (35 mL) was added (–)-α-pinene (Aldrich >91% ee, 18.4 mL, 115 mmol) dropwise at a rate that resulted in a gentle reflux. After cooling to rt, TMEDA (3.7 mL, 25 mmol) was added. The resulting mixture was heated to reflux for 30 min and then cooled to room temperature. The reaction mixture was stored in a freezer (–20 °C) for one day. The supernatant was removed by cannula and the solid was washed with cold pentane (2 x 30 mL). The colorless solid was dried under vacuum.

To a solution of this $(IpcBH_2)_2$-TMEDA complex (9.3 g, 24 mmol) in THF (30 mL) was added boron-trifluoride etherate (5.6 mL, 46 mmol) dropwise over 3 min and the mixture was stirred at room temperature for 2.5 h. The resulting slurry was filtered into a 3-neck 500 mL flask and THF washes of the residue (2 x 15 mL) were added to the filtrate. The flask was cooled to -25 °C and a solution of **6b** (8.2 g, 33 mmol) in THF (10 mL) was added dropwise. The resulting mixture was stirred at -25 °C for 8 h and then stored in a freezer (–20 °C) for 2 days. Methanol (4 mL) was added at -25 °C (evolution of H_2 gas) and the mixture was warmed to room temperature. Sodium hydroxide (11 mL of a 3M aqueous solution, 33 mmol) was added, followed by addition of 30% H_2O_2 (12 mL). The mixture was warmed to 50 °C and stirred for 1.5 h. After cooling, the aqueous layer was extracted with diethyl ether (3 x 50 mL), the combined organics were washed with water (30 mL) and brine (30 mL) and dried over $MgSO_4$. Concentration and purification by flash chromatography (1:9 ethyl acetate/hexanes) gave alcohol **7** (7.51 g, 27.9 mmol, 85%) as a colorless semi-solid. Recrystallization with hexanes gave a crystalline product (mp = 48 °C, $[\alpha]^{25}_D$ = –18.6 (c 0.0013, $CHCl_3$)). Mosher ester of the recrystallized alcohol showed a single diastereomeric ester (NMR).

[4] Brown, H. C.; Schwier, J. R.; Singaram, B. *J. Org. Chem.* **1978**, *43*, 4395. Brown, H. C., Jadhav, P. K., Mandal, A. K. *J. Org. Chem.* **1982**, *47*, 5074-5083.



3 **9**

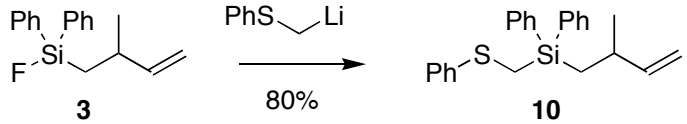
***tert*-Butyl 2-(1-(2-methyl-3-butenyl)diphenylsilyl)-pyrrolidine-1-carboxylate (9).** To a -78 °C solution of Boc-pyrrolidine (5.05 g, 29.5 mmol), TMEDA (3.61 mL, 24.0 mmol) in ether (50 mL) under argon was added dropwise *sec*-butyllithium (17.2 mL of a 1.4M solution in cyclohexane, 24.0 mmol). After stirring for 5 h at -78 °C, fluorosilane **3** (5.0 g, 18.5 mmol) in ether (20 mL) was added dropwise via cannula and the stirring was continued for additional 18h at -78 °C. After the solution was warmed to room temperature, 5% phosphoric acid (25 mL) was added and the mixture was stirred for 10 min. The organic phase was washed with 5% H_3PO_4 (2 x 5 mL), and dried over $MgSO_4$. Concentration and flash chromatography (1:20 ethyl acetate/hexanes) gave **9** as a colorless oil (6.24 g, 80%).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.64-7.59 (m, 4H), 7.42-7.33 (m, 6H), 5.70-5.63 (m, 1H), 4.76-4.65 (m, 2H), 3.98-3.93 (m, 1H), 3.39 (br s, 1H), 2.76-2.73 (m, 1H), 2.04-1.96 (m, 1H), 1.80 (m, 1H), 161.147 (m, 2H), 1.40 (s, 9H), 1.34-1.17 (m, 2H), 0.88-0.79 (m, 3H)

^{13}C NMR (100 MHz, $CDCl_3$) δ 154.9, 146.7, 135.9, 135.8, 134.7, 129.3, 127.7, 110.9, 78.7, 46.7, 33.6, 28.6, 28.3, 25.3, 23.6, 23.4, 20.4

IR (neat) cm^{-1} 3070, 2974, 2875, 1689, 1427, 1109, 907

Exact mass (FAB) $(M + Na)^+$ calc. for $C_{26}H_{35}NO_2SiNa$ 444.2334 found 444.2338



3 **10**

1-(2-methyl-3-butenyl)-diphenyl-(phenylthiomethyl) silane (10). To a 0 °C solution of thioanisole (250 mg, 2 mmol) in THF (3 mL) was added dropwise *n*-butyllithium (1.1 mL of a 2M solution in hexanes, 2.2 mmol) over 5 minutes and the resulting mixture was stirred for 2 hours. This solution was added via cannula over 5 minutes to a –78 °C solution of fluorosilane **3** (200 mg, 0.738 mmol) in THF (6 mL). The reaction mixture was warmed to room temperature over a period of 4 h. After addition of saturated ammonium chloride (10 mL), the aqueous layer was extracted with diethyl ether (4 x 20 mL), the combined organics were dried over $MgSO_4$ and concentrated. Purification by column chromatography gave **10** as a colorless oil (300 mg, 80%).

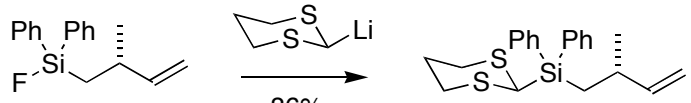
Rf = 0.85 (5:95 ethyl acetate/hexanes).

^{1}H NMR (400MHz, $CDCl_3$) δ 7.52-7.16 (m, 15H), 5.68 (m, 1H), 4.80-4.68 (dd, J = 15.2, 11.6 Hz, 2H), 2.68 (s, 2H), 2.37 (m, 1H), 1.43-1.33 (dd, J = 7.2, 15.2 Hz, 1H), 1.26-1.21 (dd, J = 6.8, 15.8 Hz, 1H), 0.92 (d, J = 6.8 Hz, 3H).

^{13}C NMR (100MHz, $CDCl_3$) δ 146.5, 135.4, 130.0, 129.0, 128.2, 128.27, 126.7, 125.2, 112.0, 34.2, 24.4, 20.9, 16.4.

IR (neat) 3069, 2958, 2899, 1586, 1479, 1427, 1111, 997 cm^{-1}.

Exact mass $(M + Ag)^+$ Calcd for $C_{24}H_{26}SSiAg$ 481.0575, found 481.0509



3 **11**

2-(1-(2*R*)-(2-methyl-3-butenyl)diphenylsilyl)-1,3-dithiane (11). To a solution of 1,3-dithiane (165 mg, 1.38 mmol) in 7 mL of THF at –78 °C was added TMEDA (0.38 mL, 2.5 mmol) dropwise followed by *sec*-butyllithium (0.90 mL, 1.4M, 1.26 mmol). The mixture was stirred at –25 °C for 2 h and cooled to –78 °C. A solution of fluorosilane **(*R*)-3** (310 mg, 1.15 mmol) in THF (3 mL) was added dropwise via a cannula. The mixture was stirred at –20 °C for 3.5 h and then allowed to gradually warm to room temperature overnight. The mixture was diluted with saturated NH_4Cl (5 mL) and the organic phase was extracted with ether (3 x 15 mL). The combined organics were washed with water, dried over $MgSO_4$ and concentrated. Purification by flash chromatography using (1:30 ethyl acetate/hexane) gave **11** as colorless oil (350 mg, 86%).

R_f = 0.40 (1:25 ethyl acetate/hexane)

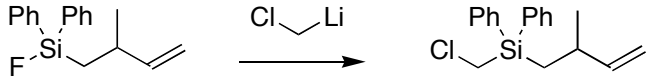
$[\alpha]_D$ –2.3 (*c* 0.5, $CHCl_3$)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (m, 4H), 7.36-7.46 (m, 6H), 5.70-5.79 (ddd, *J* = 7.2, 10.4, 17.2 Hz, 1H), 4.80-4.85 (ddd, *J* = 1.2, 1.6, 17.2 Hz, 1H), 4.74-4.77 (ddd, *J* = 0.8, 1.6, 10.4 Hz, 1H), 4.25 (s, 1H), 2.87-2.94 (m, 2H), 2.68-2.73 (m, 2H), 2.40-2.44 (m, 1H), 2.02-2.08 (m, 2H), 1.45 (dd, *J* = 6.8, 15.2 Hz, 1H), 1.31 (dd, *J* = 7.0, 15.2 Hz, 1H), 0.94 (d, *J* = 6.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 146.1, 135.8, 132.6, 132.4, 129.8, 127.6, 111.3, 33.6, 33.0, 31.6, 26.0, 23.7, 19.3.

IR (neat) 3070, 3050, 2956, 2902, 2866, 1427, 1265, 1110, 998, 909, 737, 700 cm^{-1}.

Exact mass (FAB+) Calcd for $C_{21}H_{25}S_2Si$ 369.1158 ($M-H^+$), found 369.1167.



3 **12**

Chloromethyl-1-(2-methyl-3-butenyl)diphenylsilane (12).

To a -78 °C solution of fluoro(2-methylbut-3-enyl)diphenylsilane **3** (500 mg, 1.6 mmol) and bromochloromethane (410 mg, 3.2 mmol) in THF (8 mL) was added *n*-butyllithium (3 mL, 1.6 M in hexanes, 4.8 mmol) over a period of 10 minutes. After the addition was completed, the reaction was maintained between –70 and –60 °C for 6 h. The cooling bath was removed and the solution allowed to warm to room temperature. Concentration gave a white residue which was extracted with hexanes (3 x 20 mL). The combined extracts were concentrated and purified by flash chromatography (hexanes) to give **12** as a colorless oil (500 mg, 90%).

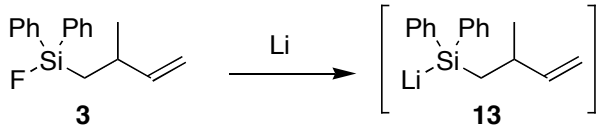
Rf = 0.6 in hexanes

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47-7.18 (m, 10H), 5.68 (m, 1H), 4.83-4.71 (m, 2H), 3.25 (s, 2H), 2.25 (m, 1H), 1.46-1.19 (m, 2H), 0.93 (d, 3H, J = 6.9 Hz).

^{13}C NMR (75 MHz, $CDCl_3$) δ 144.9, 134.0, 132.7,128.7, 126.9, 110.7, 32.7, 27.4, 23.0, 18.3.

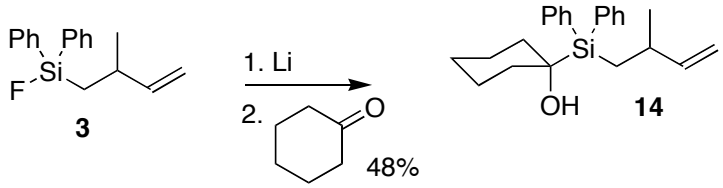
IR (KBR, neat) 3070, 2960, 2925, 1637, 1589, 1427, 1260, 1111, 700 cm^{-1}.

Exact Mass (FAB) $[M\text{-}Cl]^+$ calculated 265.1101, found 265.1401.



3 **13**

1-(2-Methyl-3-butenyl)-diphenylsilyllithium (13). Lithium granules (69.4 mg, 10 mmol) under argon in a 10 mL flask were washed with hexanes (4 x 10 mL) and then the flask was cooled to 0 °C. To this flask was added fluorosilane **3** (200 mg, 0.738 mmol) in THF (3 mL, ca. 0.25 M). The mixture was stirred at 0 °C for 12 h to give a deep red solution containing lithium fluoride. The lithium fluoride was generally removed by centrifugation.



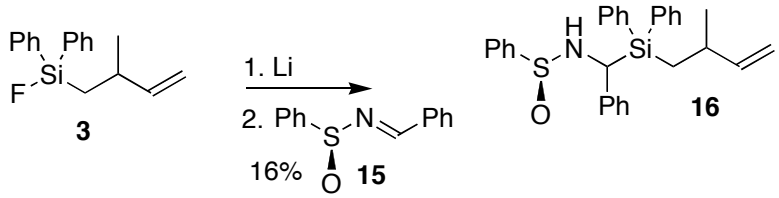
1-(1-(2-methyl-3-butenyl)-diphenylsilyl) cyclohexanol (14). To a -78 °C solution of cyclohexanone (50mg, 0.5 mmol) in THF (1 mL) was added freshly prepared silyl anion **13** (0.738 mmol) via cannula over 5 minutes. The reaction mixture was then warmed to room temperature over a period of 8 hours. After addition of saturated ammonium chloride (5 mL), the aqueous phase was extracted with ethyl acetate (4 x 20 mL). The combined organics were washed with brine (10 mL), dried over $MgSO_4$ and concentrated. The resulting yellow oil was purified by flash chromatography to give **14** as a pale yellow oil (78 mg, 48%).

R_f = 0.45 (15:85 ethyl acetate/hexanes).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52-7.15 (m, 10H), 5.81-5.72 (m, 1H), 4.88-4.78 (m, 2H), 2.41 (m, 1H), 1.78-1.54 (m, 10H), 1.24-1.12 (m, 3H), 0.98 (d, J = 6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 147.3, 134.4, 130.1, 130.13, 128.57, 128.25, 127.0, 124.92, 112.1, 77.6, 39.3, 34.0, 25.8, 24.3, 23.5, 22.5.

IR ($CDCl_3$) 3393, 3068, 3023, 2930, 2861, 1698, 1590,1447, 1428, 1260, 1115, 970, 736 cm^{-1}.



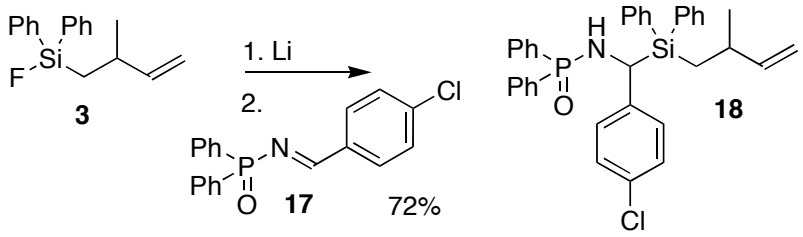
Phenyl *N*-(1-phenyl-1-(1-(2-methyl-3-butenyl)-diphenyl silyl))methyl sulfinamide (16). To a solution of sulfinimine **15** (100 mg, 0.41 mmol) in THF (2 mL) at 0 °C was added boron trifluoride etherate (50 μL, 0.4 mmol) and the resulting solution was stirred at 0 °C for 1 h. To this solution was added silyl anion **3a** (0.738 mmol, 0.25M) at -78 °C and the reaction was warmed to room temperature over a period of 12 hours. Saturated ammonium chloride (10mL) was added and the aqueous layer was extracted with ethyl acetate (4 x 10 mL). The combined organic extracts were dried over $MgSO_4$ and concentrated. Flash chromatography of the yellow oil (1:9 ethyl acetate/hexanes) gave **16** as a colorless solid (30 mg, 16%).

R_f = 0.25 (1:9 ethyl acetate/hexanes).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67-7.30 (m, 13H), 7.04-672 (m, 4H), 6.72-6.68 (m, 2H), 5.65 (m, 1H), 4.87 (t, J = 6.6 Hz, 1H), 4.79 (d, J = 6.9 Hz, 1H), 4.75-4.70 (m, 1H), 4.58 (d, 1H), 4.51 (d, 1H), 2.31 (s, 3H), 1.20-0.94 (m, 2H), 0.90 (d, J = 6.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 146.5, 136.5, 136.4, 130.3, 129.2, 128.4, 128.2, 128.03, 128.0, 127.8, 126.0, 125.8, 111.9, 76.6, 33.8, 22.0, 21.5.

IR (neat) 3262.43, 3068.30, 2923.3, 2855.7, 1558.8, 1427.6, 1108.2, 1069.1, 998.5 cm^{-1}.



Diphenyl *N*-(1-(4-chlorophenyl)-1-(1-(2-methyl-3-butenyl)-diphenylsilyl))methyl phosphinamide (18). To a solution of phosphinimine **17** (135 mg, 0.4 mmol) in THF (2 mL) at -78 °C was added silyllithium **12** (0.738 mmol, 0.25 M in THF) and the reaction was monitored by TLC. The reaction was allowed to warmed to room temperature over a period of 12 hours. After addition of 5% acetic acid in methanol (5 mL), the mixture was extracted with ethyl acetate (3 x 20 mL), washed with water and brine (10 mL), dried over $MgSO_4$ and concentrated. Purification by flash chromatography gave **18** as a solid (mp = 148-150 °C, 386 mg, 72%).

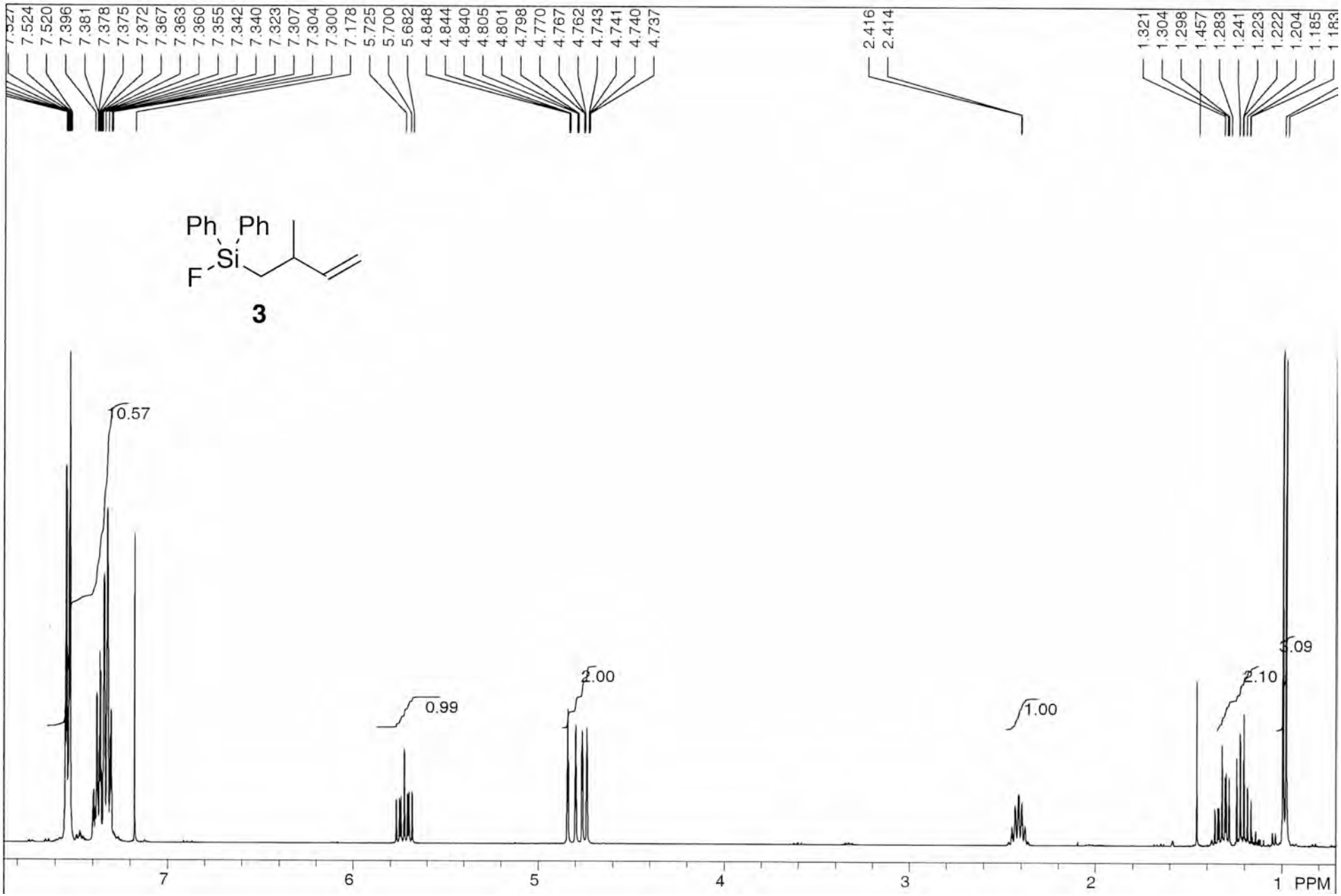
R_f = 0.54 (7:3 ethyl acetate/hexanes).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58-7.09 (m, 22H), 6.93 (d, J = 8.4 Hz, 2H), 5.51 (m, 1H), 4.60-4.50 (m, 2H), 4.34 (m, 1H), 3.25 (m, 1H), 2.20 (m, 1H), 1.05 – 0.83 (m, 2H), 0.75-0.69 (dd, J = 6.7 Hz, 10.2 Hz, 3H)

^{13}C NMR (100 MHz, $CDCl_3$) δ 146.30, 136.46, 132.77, 132.29, 132, 131.91, 131.70, 130.57, 130.39, 129.08, 128.77, 128.36, 128.23, 128.09, 112.06, 111.85, 33.80, 24.11, 23.66, 19.51

IR ($CDCl_3$) 3374, 3175, 3051, 2916, 2866, 1498, 1436, 1190, 1122, 1067, 910 cm^{-1}.

Exact mass (TOF) (MH+) calc. $C_{36}H_{35}ClNOPSi$ 592.1914, found 592.1967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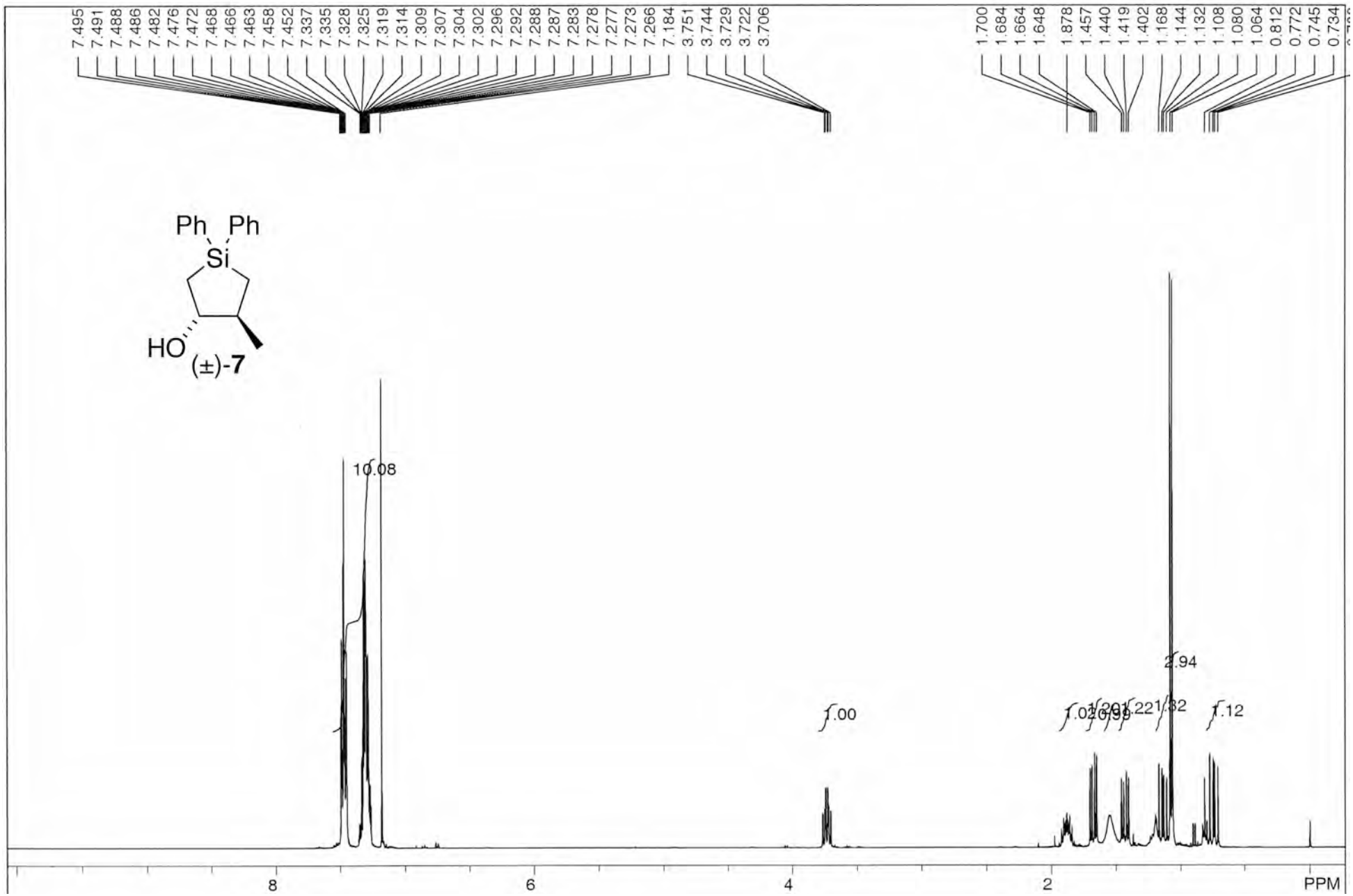
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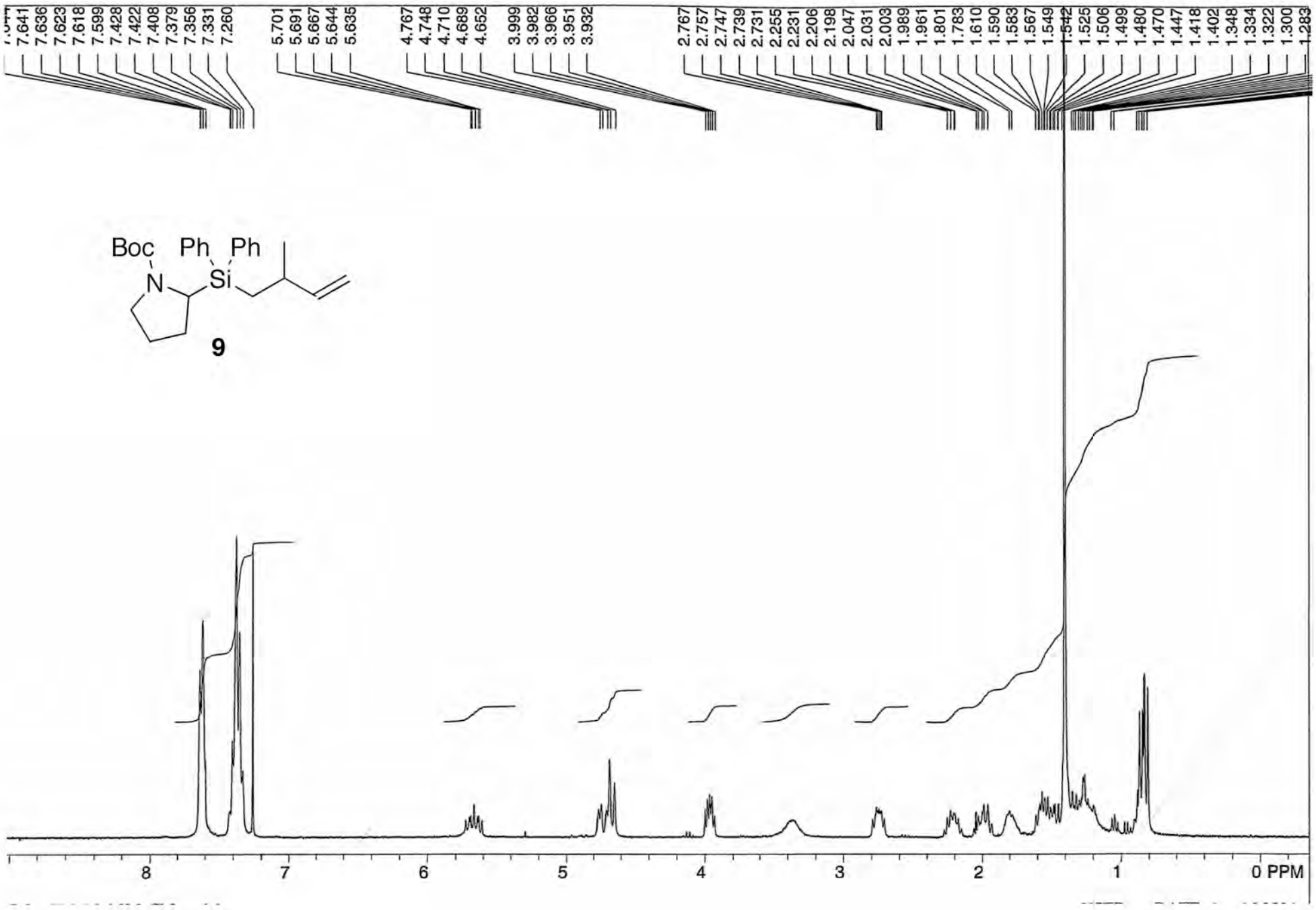
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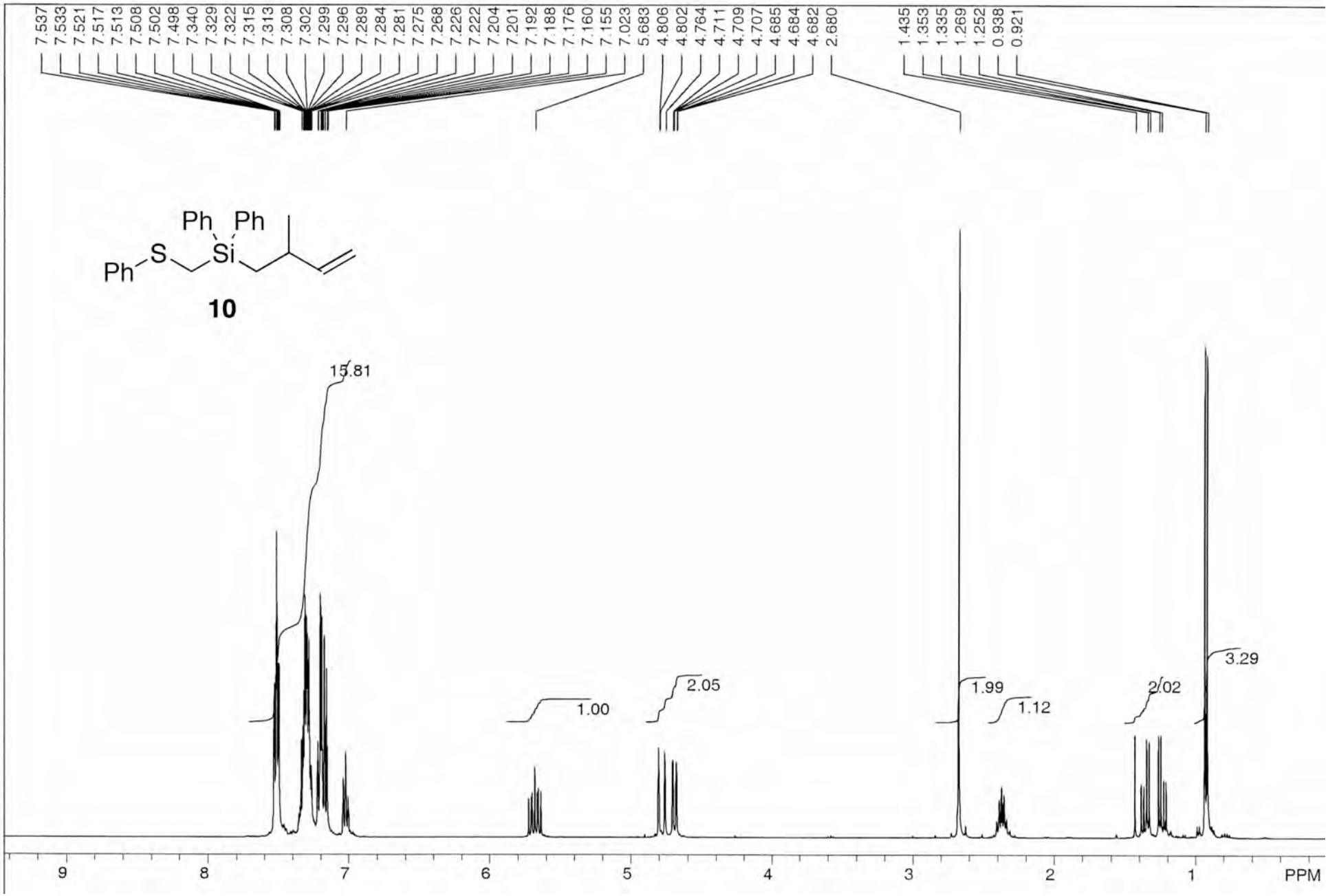
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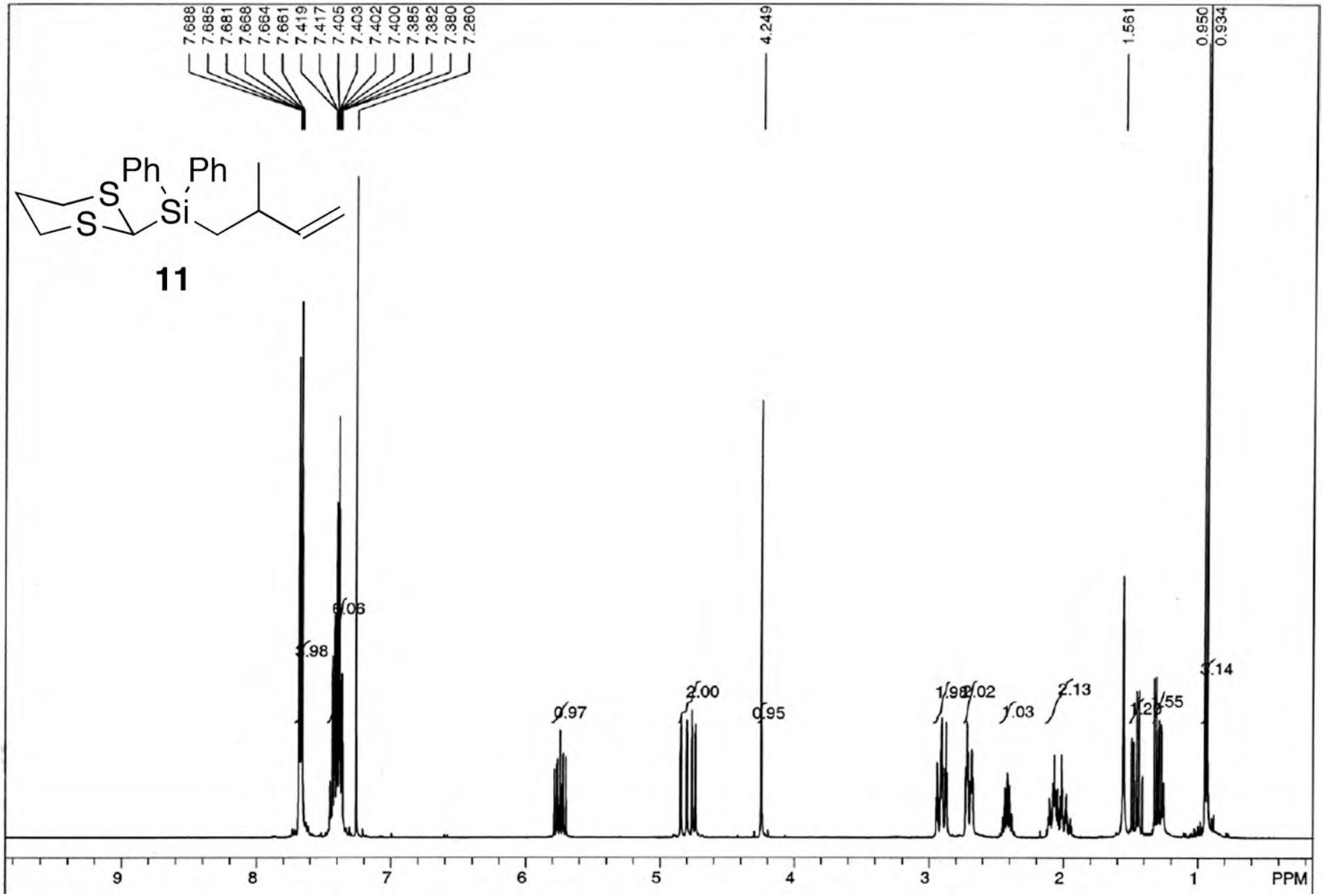
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S
Si
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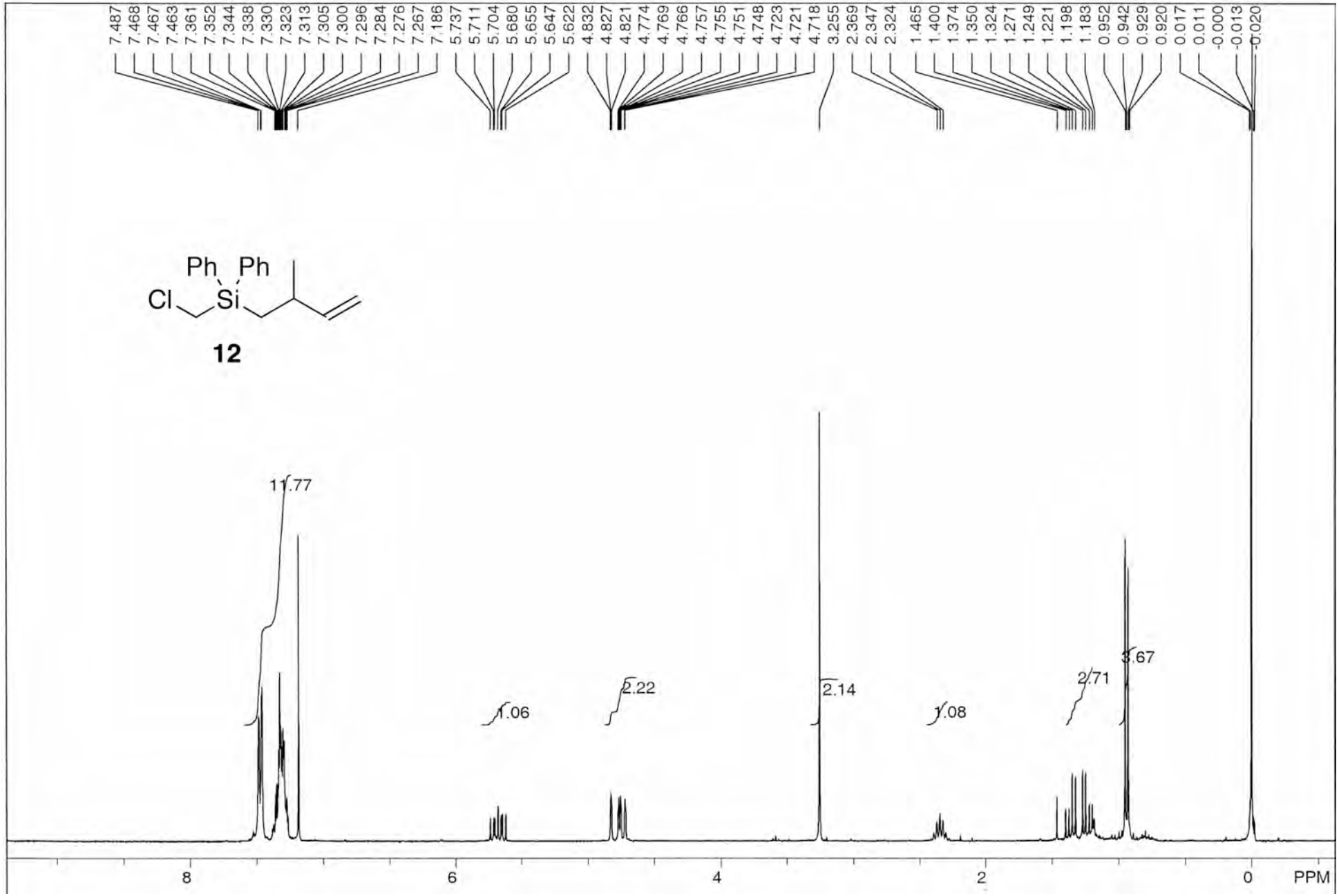
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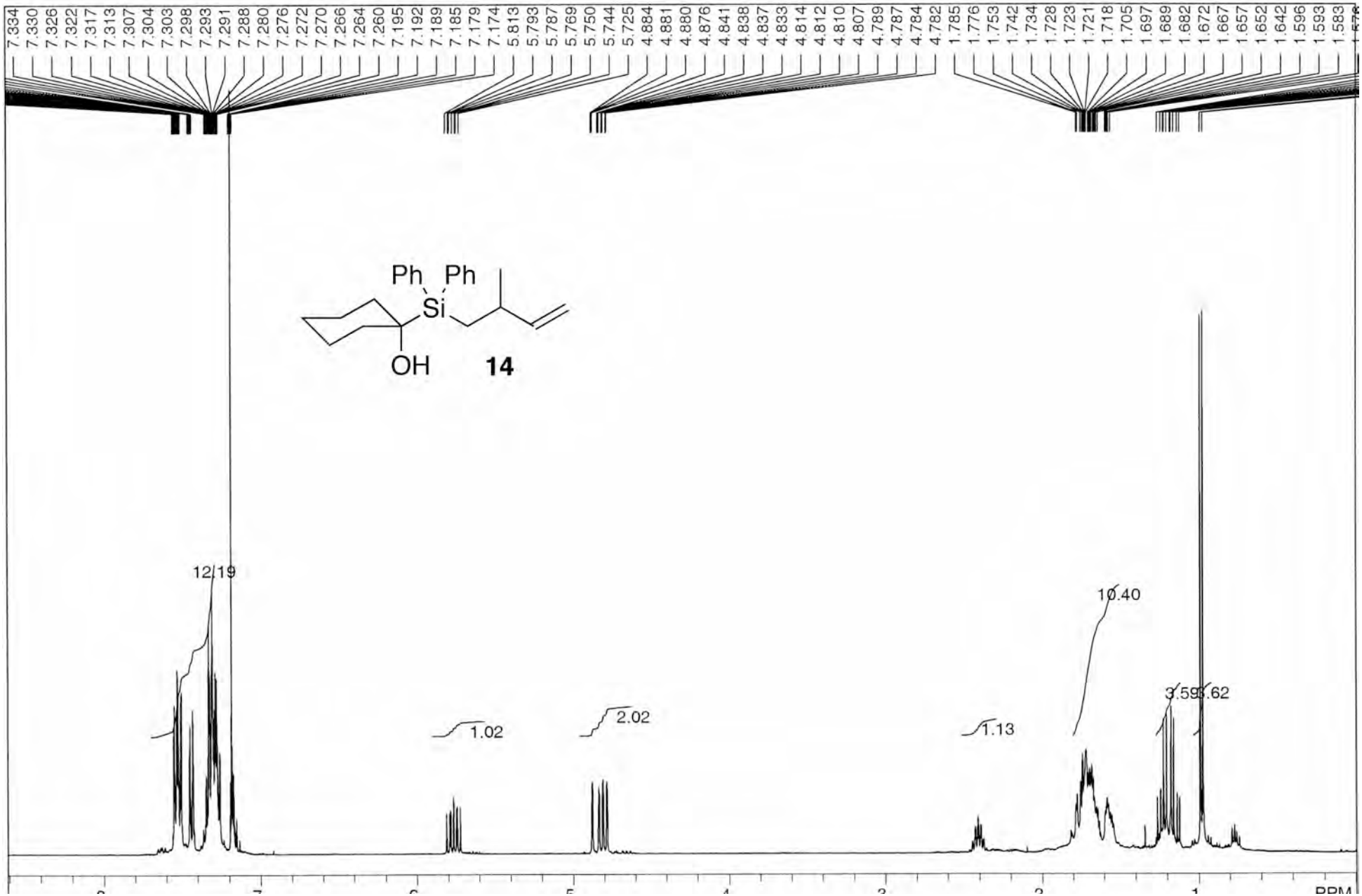
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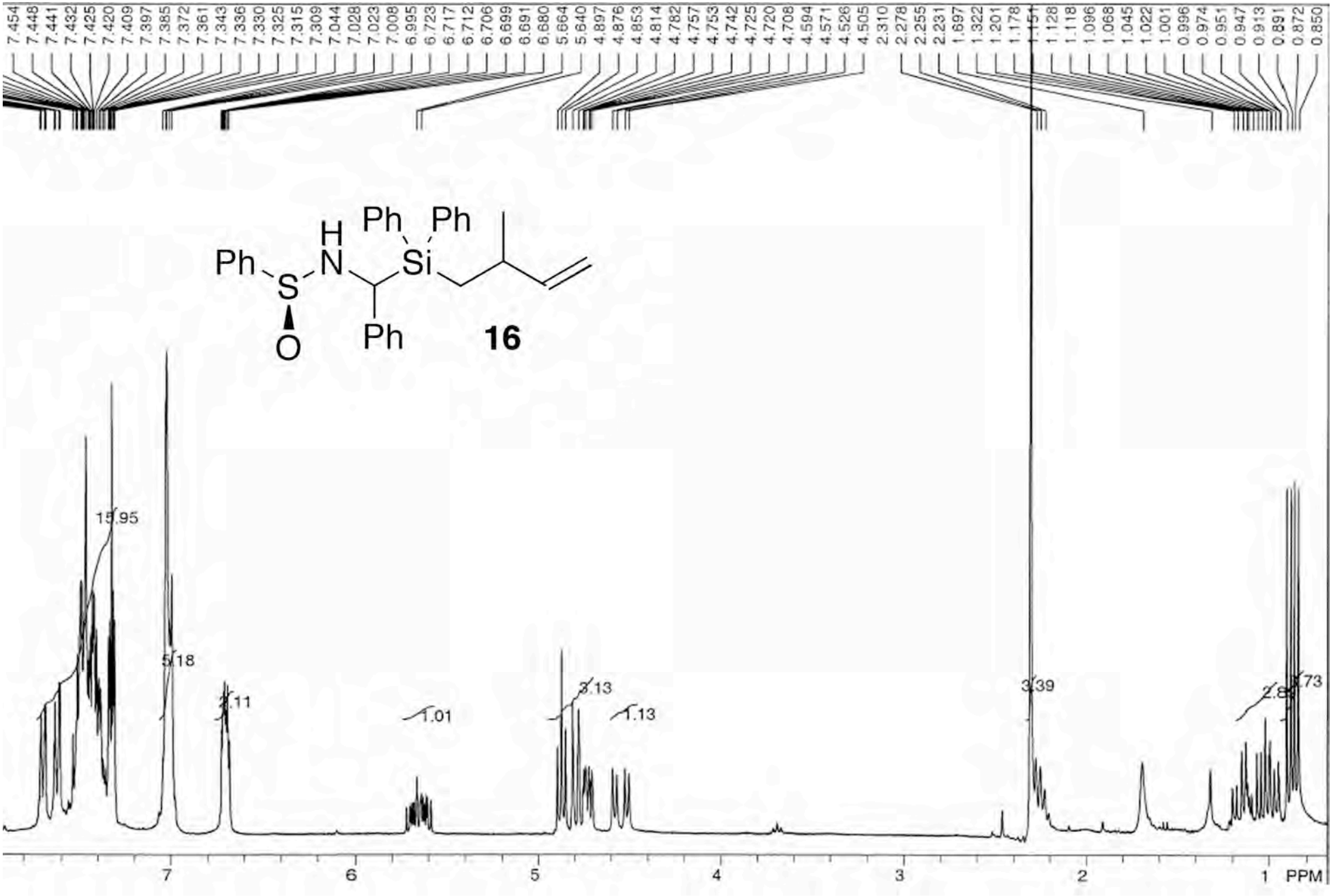
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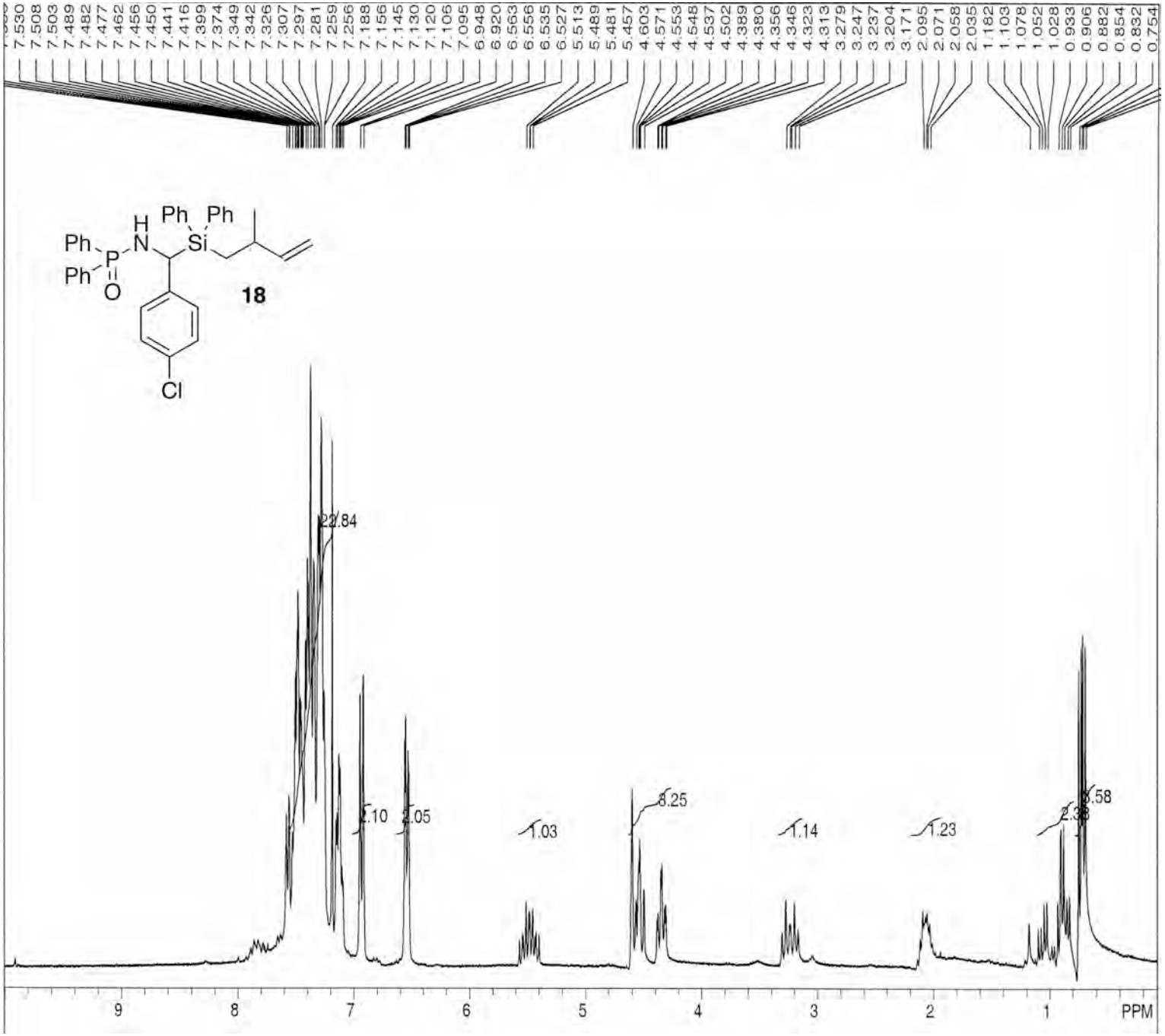
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